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# GRAPHITE

ITS

Properties, Occurrence, Refining and Uses

BY

FRITZ CIRKEL, M.E.

OTTAWA, CANADA,

## DEPARTMENT OF MINES MINES BRANCH

HONOURABLE WILLIAM TEMPLEMAN, MINISTER.

EUGENE HAANEL, PH. D., DIRECTOR OF MINES.

## **GRAPHITE**

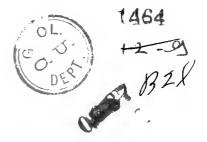
ITS

Properties, Occurrence, Refining and Uses

BY

FRITZ CIRKEL, M.E.

OTTAWA, CANADA 1907



Montreal, July 1, 1906.

Dr. Eugene Haanel, Esq.,

Director of Mines,

Ottawa, Ont.

Sir,—I have the honour to submit herewith my report on the graphite resources of the Dominion, together with a synopsis of what is generally known regarding graphite occurrences in foreign countries.

I have added also a treatise on the subject of refining, and of the uses of the mineral, based on a personal inspection and examination of the more prominent graphite refineries, pencil and crucible factories in Canada, United States, Bavaria and Austria.

I have to acknowledge with sincere thanks the valuable aid you have given me by your suggestions and kindly criticisms of this manuscript.

I have the honour to be,

Sir, your obedient servant,
FRITZ CIRKEL.

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A graphite mine in the Jungle of Ceylon.

### List of Maps Showing Graphite Locations.

#### PROVINCE OF QUEBEC.

Townships of Bouchette, Cameron and Wright.

Township of Amherst.

Townships of Buckingham and Lochaber.

County of Argenteuil. County of Ottawa.

#### PROVINCE OF ONTARIO.

Townships of Bedford, Loughborough, Crosby N., Burgess and Elmsley N.

Townships of Ashby, Denbigh, Faraday and Dungannon.

" Brougham and Blithfield.

Township of Marmora.

" Westmeath.



Canadian Graphite Ore: Pure Vein Graphite Exhibiting a Coarse Lamellar Structure,

#### INTRODUCTION

Graphite is a mineral whose distribution is world wide and whose application in the arts and industries is manifold. It is hoped that this treatise on the subject, summarizing all practical information now available regarding the mineral, will be of interest not only to those engaged in the mining or exploitation of graphite deposits, but also to those interested in this mineral or its products in a purely commercial sense. The literature on the subject is so meagre and so scattered through technical and scientific journals and Government reports, that it has been very difficult to obtain an intelligent conception of the present status of the graphite industry in all its phases.

In this monograph the writer discusses the geological occurrence of the mineral, both in Canada and elsewhere, the shape and structure of the ore bodies, their manner of development. the composition of the ores and their properties, the various methods of refining and finally the application of the purified It is believed, that the chapter dealing with the properties, geological occurrence and uses is as concise and complete an exposition on the subject, as is possible under the circumstances, but the writer readily admits that the chapter dealing with the refining is, for lack of reliable date, incomplete. Most of the graphite mill owners are reticent in giving information on the subject of refining, indeed it must be said, that this is one of the industries, on which not only is there hardly anything published, but it is difficult, if not impossible, to study the various mill schemes with the object of generalizations. Nearly every mill follows its own method, worked out and adapted to the peculiar characteristics of the ore, to meet the requirements and the exigencies of the market.

The writer therefore has confined himself to the enumeration and description of machinery for drying, crushing and concentration purposes, which so far have been adopted in the mills. He has also summarized the principles upon which the separation of graphite from the gangue is based, with an outline of several mill schemes, which are in operation on the American continent and in Europe, but he has not, for reasons just stated, gone into the description of special devices for the production of certain grades.

The writer has to express his sincere thanks to mine and mili owners and managers and to manufacturers of machinery for their courtesy in supplying valuable information for this treatise. He is also greatly indebted to Dr. Eugene Haanel, Director of Mines, Ottawa, and to Dr. J. T. Donald, of Montreal, for suggestions and kindly criticisms of the manuscript for this report.

FRITZ CIRKEL.

MONTREAL, 2nd July, 1906.

#### CHAPTER I.

## HISTORY, CHEMICAL AND PHYSICAL PROPERTIES OF GRAPHITE.

#### HISTORY.

Graphite\* was known by the ancients, for pieces of graphite and stone vessels painted with the mineral have been found in ancient graves. However this knowledge, which may not have been more than local, apparently lapsed subsequently, for we find that nowhere is mention made of its existence or application until the middle ages. Graphite is there mentioned as a mineral curiosity, but the name has been very often applied, even in recent times to similar looking minerals such as molybdenite. though the mineral has been known for such a long time, its true chemical nature was not recognized until a comparatively late date. The generally accepted opinion was, that graphite contained lead, hence the name black-lead, while in German its property of leaving a mark on paper is signified in the term "Reissblei"—drawing lead. Later chemists and mineralogists made the mistake of applying the name graphite to different minerals, with no regard to their chemical composition.

The German chemist Heinrich Pott, 1692–1777, was the first to demonstrate that graphite contains no lead, but it is uncertain whether he experimented with real graphite or with molybdenite, because like the chemists Quist (1754) and Pott, he did not distinguish between the two minerals.

Karl Wilhelm Scheele (1742-1786), was the first to recognize and to establish the true chemical nature of the two so similar looking minerals. Scheele† showed in 1779 that graphite burns to carbonic acid gas in a current of oxygen, hence concluded, that it consisted of carbon; but even long after this, noted chemists doubted the existence of carbon in graphite, since

<sup>\*</sup> From the Greek "graphein" to write; also "plumbago," black-lead; German: Pottlot, Ofenschwarz, Reissblei; French: Fer carbure, crayon noir. Carbo mineralis. As a modification of carbon it is known as  $\beta$  carbon (Beta carbon). The name plumbago seems to be derived from the Italian "grafio piombino," which like the other name graphite, from  $\gamma\rho a\phi\omega$ , I write, indicates its use.

† Dammer, Handbuch der anorg. Chemie—Bd., II part I, 260-1894.

the analytical methods for the investigation of such minerals were at that time very imperfect.

It was not until the strong resistance of graphite to all reagents was established, and the commonly accompanying impurities were recognized and removed that the true nature of graphite as pure carbon was recognized and generally accepted.

The word graphite appears to have been first used by the well known mineralogist A. G. Werner (1750-1817) and it has been generally accepted since that time.

As to the early technical application of graphite the oldest authentic records date back as far as the year 1400. We find that graphite was first used in the manufacture of crucibles in Hafnerzell near Passau on the Danube, Bavaria, the mineral being obtained from the mines in the vicinity of these places. Crucibles made of pure clay and graphite were used at that time in the laboratories of Alchymists for melting the precious metals and in their futile endeavour to find the "stone of the wise." Far more important than the manufacture of crucibles, which for centuries remained a very small industry, is the application of graphite to the manufacture of pencils, and it is principally this branch of the industry, which on account of the employment of the purer qualities stimulated the search for and the economic refining of the mineral.

Cennino Cennini\* born in Florence, Italy, about 1370, mentions in his book on painting the use of a pencil for his paintings, consisting of two parts of lead and one part of tin.

Conrad Gessner, in his work "De rerum fossilium figuris," in 1565, mentions the use of lead pencils, but these must have been a great curiosity at that time, because he held it important to add a picture of a black lead pencil and underneath is written: "Stylus inferius depictus ad scribendum factus est, plumbi cuyusdam (factitii puto, quod aliquos stimmi Anglicum vocare audio) genere, in mucronem derasi, in manubrium ligneum inserti."+

La Moine cites a document of 1387 ruled with graphite. (?)

An impetus to the actual manufacture of lead pencils was given through the discovery of the celebrated mines of Borrowdale in Cumberland, England, which occurred about 1550. Ac-

<sup>\*</sup> Traktat der Malerei, Vienna, 1888.
† "The pencil represented below is made for writing, of a certain kind of lead (which, I am told, is an artificial substance termed by some, English antimony) sharpened to a point and inserted in a wooden handle."

cording to several writers\* the new mineral was used by the farmers at Keswick (some miles distant from the mines) in marking the cattle.

It is difficult to state with any degree of accuracy where the first pencils were made. At all events it is very likely that the art loving Italians were the first ones to employ the new mineral in drawing. Its advantage over a great many other appliances at that time in use was soon acknowledged.† It appears also that the graphite for these Italian pencils had been imported from England; it was called at that time Flanders stone and although there were no graphite mines at that time in Holland, it is reported that Italy was supplied with the mineral by Flemish merchants, who in turn imported it from England.

#### CHEMICAL AND PHYSICAL PROPERTIES OF GRAPHITE.

Pure graphite like diamond and charcoal consists of carbon, but in form, color, hardness and other physical properties it is so different from these minerals that Brodiet thought it should not be considered as a modification or an allotropic orm of carbon, but as a separate element, which he termed "Graphon.' however, graphite like the diamond and amorphous carbon burns in a current of oxygen to carbonic acid gas this supposition is unfounded.

The three forms of carbon, charcoal, graphite and diamond may be distinguished by certain differences in their physical properties. Thus amorphous carbon or charcoal has a density of 1.3 to 2.0, graphite 2.1 to 2.58, diamond 3.5. Most forms of amorphous carbon when rubbed on paper leave a dull black mark, graphite leaves a mark having a bright metallic lustre, diamond leaves no mark. Amorphous carbon, if cut with a knife, is usually brittle, graphite is like a very hard wax, while diamond is untouched even by the hardest file. Diamond and most forms of amorphous carbon are very poor conductors of electricity and heat, but graphite is comparable with metals in this respect.

Graphite commonly occurs in embedded foliated masses, also in micaceous columnar, radiated, scaly, or slaty forms;

<sup>\*</sup> Robinson, Essay towards a natural history of Westmoreland and Cumberland, London, 1709, page 74.

<sup>†</sup> Terrante Imperato erato del historia naturale libre XXVIII, Napoli. 1599, page 122 and 678. ‡ Brodie, Ann. Ch. 114, 2.

occasionally it is granular and compact whilst at other times it is of a decidedly earthy texture. Crystals of graphite are very rare; they are six sided, tabular, nearly always striated, with indistinct faces. Graphite crystalizes in the Rhombohedral system. Clark, Sukow and Nordenskjold\* however, especially the latter, who made a series of accurate measurements on crystals from Pargos, Ersby and Stargard in Finnland, came to the conclusion that graphite crystallizes in the monoclinic system. Perfectly formed crystals in nature are very rare, most of them being found in granular crystalline limestone and in meteorites. To liberate graphite crystals from the enclosing rock mass is a most difficult task, owing to the most delicate and soft nature of the mineral. For this reason the question as to what system of crystallization the mineral really belongs has not been decided definitely, but observations point to the probability of its belonging to the hexagonal system and rhombohedral division. from these crystals graphite occurs, also as pseudomorphous crystals of pyrite.

So far the best crystals have been obtained from meteorites; graphite in very regular complete crystals was discovered in the meteorites from Aroa, Ungary, also in the meteorites from Prundegin (Western Australia) and Crosby creek. Fletcher believed one graphite crystal obtained from the last named meteorite to be a modification of the regular hexagonal graphite, but other mineralogists recognized the same as pseudomorphoses of graphite to diamond.

Graphite occurs in crystalline aggregates, which may be described as accumulations of imperfect crystals, far more frequently than in crystals; they are composed of small scaly lamellar particles or columnar or radiated individuals, the latter very often resembling the structure of wood. These columnar or long fibred aggregates, which consist generally of a very pure graphite, are frequently met with in nature, as a rule are in a more solid condition than the lamellar varieties and exhibit generally a regular and more even structure. The latter is usually erect and at right angles to the enclosing rock surface; frequently it is curved, as though from pressure. It breaks very often in the direction of the structure into more or less angular aggregates, each aggregate being composed of thin, narrow foliæ of very

<sup>\*</sup> Ed. Donath Der Graphit, 1904, page 3.

### PLATE II.

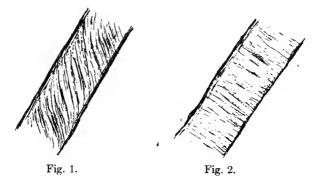


Canadian Graphite Ore: Pure Vein Graphite, Exhibiting a Radiated Structure,

uniform width. The length of the columns varies from one half to ten centimeters. Foreign mineral matter is often distributed through the structure and appears as a very fine film upon the graphite.

Graphite occurs very frequently in a dense or earth like condition in compact masses, and in this state it is very soft and easily disintegrated.

Natural graphite therefore shows partly the lamellar condition and is less friable or it entirely lacks the definite crystalline structure and is then very soft and more friable. In the former case



Figs. 1 and 2.—Columnar or Long Fibred Aggregates of Graphite.

we have the "scaly," "lamellar" or "flaky" graphite, in the latter the "amorphous," "compact" or "earthy" graphite.

Graphite shows a perfect basal cleavage similar to mica. Thin laminæ have a diamond like metallic lustre when pure. Their color varies from this to dull and earthy, according to the percentage of impurities. They have an unctuous aspect, feel greasy and slippery, are flexible but not elastic. The color of dense aggregates is iron black to dark steel gray, sometimes with opaque appearance. On larger lamellæ on account of the great lustre the colors change from a dark black to a blue gray.

Occasionally finely granular vein graphite, like that from Warrensburg,\* New York State, breaks easily into rectangular masses, which exhibit on certain of the fractured surfaces, a peculiar finely waved aspect, due to a structure, which may be described as consisting of layers of a millimeter or less in thickness, tolerably regular, and made up of minute and narrow lamellæ,

<sup>\*</sup> Geology of Canada, 1856, page 219.

arranged at right angles to the layers, and presenting a fibrous or columnar aspect when broken across. When the fracture is with the layers, and thus exposes only the ends of the lamellæ, a granular surface is presented. Fracture at right angles with the layers shows an undulating surface, recalling that of certain waved maple woods and due to the fact that the fibres of the successive layers are not quite parallel with each other.\*

Graphite is one of the softest minerals, its hardness lies between 1—2 Mohs scale. It leaves a black mark on paper and owing to this quality it is used so extensively in the manufacture of pencils. It can be readily distinguished from molybdenite, which produces a grayish green mark.†

Graphite even in its thinnest laminæ is completely opaque, however for the Xrays it belongs like the diamond to the most transparent bodies. If plates of the mineral, which contain foreign enclosures, are exposed to the influence of X rays, the enclosures appear distinctly as dark shades in the otherwise lighted background.

The specific gravity of graphite varies from 2.015 to 2.583, and this considerable variation is due to the fact that almost all natural graphites carry more or less impurities, very often consisting of iron oxide. In the following table 1 the specific gravities of several graphites are given:—

<sup>\*</sup> This Laurentian graphite, according to Prof. Chandler's analysis, consists of carbon 64.06, carbonate of lime 32.00, the remaining three per cent. being chiefly silica and iron oxide. The carbonate of lime is invisibly diffused through the mass, which effervesces freely with acids. It is not in any way connected with the peculiar waved structure, since the graphite from the famous mine of Marinski, in the Government of Irkutsk in Siberia, which presents a structure precisely similar, contains no carbonate of lime, and only small amounts of earthy impurities, amounting, according to Dumas, to only 3.7 per cent. in the purest specimens.

<sup>†</sup> Molybdenite or molybdenum sulphide is often mistaken for graphite, which it nearly resembles; it occurs in hexagonal plates or masses, or thin foliated leaves like graphite. Color is pure lead gray; streak the same; leaves a trace on paper, but not so strong as graphite. Composition: sulphur, molybdenum; infusible; differs from graphite in its paler, more metallic, tin-like color, also in giving off sulphur fumes when heated in a closed tube, also by solubility in nitric acid; occurs in granite gneiss, mica schist, not unfrequently associated with the precious ores. See also page 128.

TABLE I.

Locality.	Kind.	Spec. Gr.	Authority.
Buckingham, Canada	Columnar	2.2689	Dr. Hoffman,*
" " "	"	2.2679	"
	ronated	2.2714	**
Grenville	Columnar	2.2659	"
Ceylon	"	2.2671	44
		2.2664	"
"	Columnar	2.2546	"
"			4.6
		2.2599	11
Ticonderoga, U.S.	"	2.2647	44
Ceylon I		2.257	C. Rammelsberg†
Borrowdale		2.286	11
Upper Jenisei	• • • • • • • • • • • • • • • • • • • •	2.275	"
Upper Jenisei		2 298	46
Arendale		2.321	44
Ticonderoga		2.17	44
Ceylon II		2.246	
Furnace Graphite.		2.30	

For refined graphites lower specific gravities were found, in one case as low as 1.802.

Portions of the specimen of graphite sent from Canada to the Exhibition at London in 1862, were furnished to Mr. Regnault, the eminent French chemist and physicist, who made use of them in an investigation on the specific heat of this form of carbon. Incidental to this inquiry they were submitted to a careful analysis by Mr. Cloez. After being calcined to expel any traces of moisture, they were burned in a current of dry oxygen. The results showed, as already suspected by Regnault, that a portion of hydrogen entered into their composition and is only separated by prolonged ignition in a current of dry chlorine, which at the same time separates the earthy impurities, in the form of chlorides, and leaves the graphite as almost chemically pure carbon.‡

The specific heat of graphite is higher than that of diamond; it is for natural graphite 0.2019 and for furnace graphite 0.1970.

In the following table 2 the specific heat of natural and arti-

<sup>\*</sup> Geology of Canada, 1876-77, page 507.

<sup>†</sup> Ed. Donath, Ibid, page 4-5.

<sup>1</sup> Ann. de Chim. et de Phys. 4) VII., 450.

ficial graphites and also the percentage compositions of carbon and hydrogen are given:-

Locality.		Kind.	Carbon.	Hydro- gen.	Nitro- gen	Ash	Spec. Heat.
Buckingham,	Can	Natural " " Artificial " " "	86.8 76.35 98.56 99.5 89.51 96.97 99.1	0.50 0.70 1.34  0.60 0.76 0.39	1.87	12.6 23.4 0.2 0.68 10.4 0.4 0.79	0.1986 0.2019 0.1911 0.1977 0.2000 0.1968 0.2000

TABLE 2.

The specific heat increases beyond a temperature of 1000 and according to the following formula:-

$$C_o^t = 0.355 + 0.000006.t$$
 (Vieille.)

The heat of combustion for natural graphite was determined by Favre and Silverman at 7796 Cal. For artificial graphite from furnaces 7762.3 Cal., Berthelot and Petit found 7901.2 Cal.

The coefficient of linear expansion of graphite is at 40°C. The elongation of a unit of length from 0° to 100°C. is 50786. Graphite conducts heat better than diamond and is also known to be a good conductor of electricity. If the thermal conductivity of silver at 0° is 100, it is for pure Ceylon graphite at 22°C. 0.0693, for pure Bavarian graphite 0.20395 and a mixture of both gives 0.0346. According to Matthiessen the conductivity of refined graphite is 18 times greater than that of natural graphite.

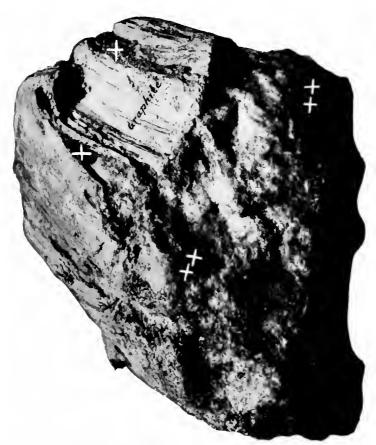
According to Muraoka\* graphite from Siberia has a conductivity at 0° compared with quick silver at 0° of 8196 x 10, Fabers pencils of 1051 x 10, retort coal of 1360 x 10, retort coal from Goudoin of 1813 x 10, carbon bar from Dubosco 2880 x 10. carbon bar from Carre 1348 x 10.

Streintz† determined for graphite in powder form a specific electrical resistance of 14.20 Ohms. According to Muraoka's tests the same is 12.20 Ohms.

Aharden‡ determined the specific resistance of a graphitised electrode of American best quality for 1 sq. millimeter

<sup>\*</sup> Wiedem. Ann. 13, page 30, 1881.

<sup>†</sup> Sammlung electrotechnicher Vorträge Stuttgart. ‡ Electrot. Zeitschr, 1901, page 584.



Canadian Graphite Ore: Pure Vein Graphite Exhibiting a Woodlike or Columnar Structure.  $\times$  Graphite.  $\times$  Limestone.

and 1000 millimeter long at 12 Ohms, and the conductivity at 0.0083.

Zellner determined for Ceylon graphite a specific resistance (at about 20°C) of 2 to 8 Ohms, for graphite electrode of Acheson 12 Ohms.

#### CHEMISTRY OF GRAPHITE.

Graphite undergoes no change when heated with exclusion of air. Its combustion in air can be effected only with great difficulty, while it burns more or less quickly in a current of oxygen. According to Moissan some varieties burn at the following temperatures in oxygen:—

Graphite from Ceylon at 665°C.

From Bohemia, Schwarzbach at 620°C.

Artificial graphite from Sugar carbon at 660°C.

"crystallized from Platinum at 575°C.

According to Gustave Rose the crystalline variety is more difficulty combustible, whereas the compact variety burns more readily than the diamond. By the addition of finely divided metallic silver the combustion is facilitated (Stolba.)

Heated in a glass tube under the influence of the blowpipe, graphite sometimes gives off an appreciable amount of water. The pure mineral is unaffected by strong mineral acids, by heating in a current of chlorine or by treating it with dilute nitric or sulphuric acid; these reagents can therefore be employed for the elimination of the accompanying impurities. It remains unchanged by fusion with caustic soda or potash. In fusing with saltpetre several varieties of graphite are oxidized according to Rammelsberg, others remain unaffected. Graphites, which burn with saltpetre, are those of Ceylon I, Borrowdale, Jenisei, Upernivik, (Greenland), Arendal. Graphite from Ticonderoga, Ceylon II and furnace graphite do not burn with saltpetre.

By heating graphite with chromic acid or with a mixture of bichromate of potassium and sulphuric acid it is burned to carbonic acid gas.

Graphite in its chemical relations occupies a position totally different from that of all other forms of carbon; amongst themselves, indeed, the several varieties exhibit differences of a remarkable character when they are acted upon by oxidizing agents.

When finely powdered graphite is heated with a mixture of one part of nitric and four parts of strong sulphuric acid, or when a mixture of fourteen parts of graphite and one part of potassium chlorate is warmed with seventy-eight parts of strong sulphuric acid, the graphite assumes a purple tint, but on subsequent washing it returns to its original colour. It is, however, no longer graphite, but contains in addition oxygen, hydrogen and sulphuric acid. When this compound is heated to redness it swells up with a copius evolution of gas and then falls to an extremely finely divided powder of pure graphite, which has a specific gravity of 2.25.

This process is employed for the purpose of purifying natural graphite. With this object it is first ground and the powder well washed in troughs in order to remove as much as possible of the earthy matter with which it is contaminated. The graphite thus obtained is pure enough for many uses; but if it is required in a very pure state, the powder must be treated with potassium chlorate and sulphuric acid as above described. The fine powder is then thrown upon the water, on the surface of which it floats, while the earthy matters sink to the bottom. The foliated graphite answers best for this purpose, the amorphous graphite being more difficult to purify. This variety may, however, also be purified, if a small quantity of fluoride of sodium be added to the mixture as soon as the evolution of chlorine trioxide gas has ceased, the object of this addition being to remove the silica as silicon tetrafluoride.

If certain graphites are heated with nitric acid, they swell up considerably and produce round, longitudinal, wormlike forms having a circumference of from one quarter to one half inch and a length sometimes of several inches. These forms are steel gray, of metallic lustre, are bent and twisted in regular curves and appear otherwise very characteristic on account of this regular structure. Fig. 3. The latter on closer examination consists of a number of oblong cells, densely grouped together, exhibiting on the outer surface long parallel folds and wrinkles. These forms are,—and this is very important,—very light in weight, and when put in water or alcohol always rise to the surface, even when saturated thoroughly with the liquid. They are as a rule very plastic, can be put into any form and can easily be compressed between the fingers. Their interior exhibits planes of a highly metallic lustre, which, according to Donath, are supposed to represent a lamellar

crystallization. This characteristic behaviour of certain graphites was first noted by Brodie, and the above process, carried out on a larger scale, has been used for the production of a graphite of exceptional purity and of a very fine and equal division (Brodie's graphite.)

Luzi and O. Lüdecke\* use this nitric acid reaction for a differentiation or classification of graphites. They hold that the graphites occur as varied modifications of carbon and divide them accordingly into two classes, those which yield to the above reaction and those which are indifferent to the same. The former kind Luzi designates as "graphite", the latter as "graphitie." In order to demonstrate this on a small scale, some finely granulated (not powdered) graphite is moistened with Nitric Acid of a



Fig. 3.

Fig. 3.—Flatulent Forms of Graphite produced by Chemical Treatment.

specific gravity of 1.52—1.54, in a platinum dish and then heated. It will then be noticed that the mineral either remains indifferent or it will expand in volume, forming those wormlike structures, sometimes of a length of five inches. On a large scale this process can be carried out in iron retorts, the acid will finally be driven off, while the wormlike structures will remain; the latter are collected and washed through troughs and the result is a graphite of great purity and fine division. Based upon this reaction Luzi, Moissan and Sestini divide the natural graphite occurrences of the world into two groups:—

Group 1.—"Graphites" yielding to the nitric acid reaction. To this group belong the following localities:—

Ceylon, Ticonderoga, Amity (New York State). Grenville, and Buckingham, Province of Quebec; Borrowdale, England; Monte Rosa, Calabria; Bamle Skütterüd, Norwegia; Marbach, Lower Austria; Pfaffenreuth, near Passau, Bavaria; Spanish graphites.

<sup>\*</sup> Thonindustric Zeitung, 1895, No. 8.

Group 2.—"Graphitites" remaining indifferent to the nitric acid reaction. To this group belong the following localities:—

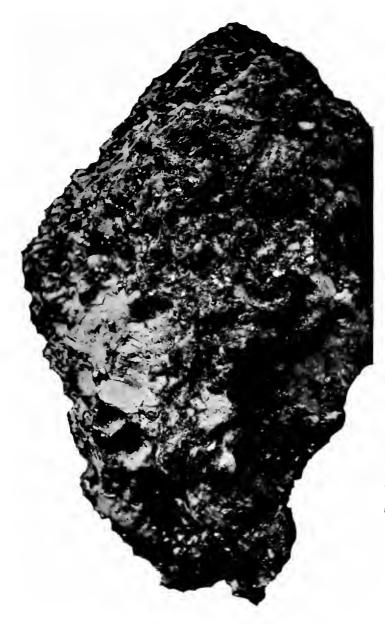
Altstadt, (Moravia); Krumau, Schwarzbach, Mugrau (Bohemia); Passau (Bavaria); Saxonian graphite; Irkutsk, Tunguska (Siberia); Storgard, (Finland); Karsock and Omesnack, (Greenland); Colfax county, (New Mexico); South Australia; Takaschimiza, (Japan); Lerigliani, Monte Pisano, Italy; all artificial graphites made by electric processes.

Weinschenk\* does not believe that "graphite" and "graphitite" are different modifications of carbon. He attributes the different behaviour of graphites in nitric acid to their different structure; he finds that the graphite, which expands so much in volume by the nitric acid reaction, consists as a rule of very fine, minute scales and plates, which on account of their capillary structure suck up the acid and swell up through the gases produced therefrom under the influence of heat. Weinschenk holds that these two kinds of graphites, designated as "graphite" and "graphitite" by Luzi, are different only by their mode of division but are otherwise strictly identical.

Weinschenk thinks also that the term "amorphous" is not applicable to graphite, since it refers to varieties, which are characterized by a very compact dense structure, but occur otherwise like the larger lamellar aggregates and consist of very minute crystalline individuals, hence cannot be considered amorphous in a mineralogical sense. Graphite occurs indeed very often in a state of very fine division, and it is difficult to recognize sometimes its crystalline structure, even with the aided eye, but in all cases the limit between crystallized and amorphous carbon is so decidedly distinct and sharp, that a transition from one state into the other is out of the question.

In this connection it may be said that in the year 1870 the celebrated French chemist M. Berthelot undertook an exhaustive investigation of the different forms of carbon. Before this work was done, there was no clear distinction between amorphous carbon and graphite. Thus coke obtained from oil, charcoal and lampblack, that had been exposed to a high temperature, retort carbon, carbon that had been obtained by calcining sugar, etc., were identified with graphite. M. Berthelot, however, devised a method of distinguishing amorphous carbon from graphite.

<sup>\*</sup> Zeitschrift der Krystallographie, 28, 291.



Canadian Graphite Ore: Disseminated Flake Graphite in Crystalline Limestone.

using for this purpose a reaction originally discovered by Sir Benjamin Brodie. Weinschenk, in support of his theory, uses Brodie's reaction, and shows that in certain properties graphite differs remarkably from the other modifications of carbon. reaction consists of treating carbon with concentrated nitric acid and potassium chlorate. Amorphous carbon is dissolved readily to a brown liquid, whereas graphite undergoes a gradual change into a yellow transparent, scaly substance, which is insoluble in nitric acid and which represents an oxidation product of graphite. This always crystalline product, which is obtainable only from graphite and not from amorphous carbon, is termed Graphitic Acid, and the production of this graphitic acid is the safest means of identifying graphite, we know at the present time.

Sir Benjamin Brodie,\* above referred to, already has 'shown, that when acted upon by certain oxidizing agents, graphite is converted into a compact substance, which contains oxygen and hydrogen and possesses the property of an acid. In order to prepare graphitic acid, an intimate mixture of one part of purified graphite and three parts of potassium chlorate is treated with so much concentrated nitric acid that the mass becomes liquid. then heated from three to four days on a water bath. The solid residue after having been washed with water and dried at 100°C is subjected four or five times to a similar treatment until no further change is observed. Graphitic acid is a stable yellow substance, existing in thin microscopic crystals, which have the property of reddening moistened blue litmus paper and are slightly soluble in pure and insoluble in acidified water. The salts have been, as yet, but slightly investigated. Graphitic acid retains in all cases the form of the original graphite as small scaly particles of the mineral, gradually attaining a yellow color and becoming to some degree transparent. Neither charcoal nor diamond yield similar compounds, and Brodie believes that graphite may be considered to be a peculiar radical, to which he gives the name of "graphon."

According to Berthelot only natural graphite forms the above compound; whilst iron graphite, as well as that found in the Cranbourne meteorite yields a chestnut brown powder on similar treatment.† Diamond is not attacked by this oxidizing mixture whilst ordinary charcoal is converted into a brown mass soluble in

<sup>Phil. Trans. 1859, page 249.
Comptes Rendus LXVIII, 183, 259, 334, 392 and 445.</sup> 

water. Berthelot has made use of this property for the purpose of estimating the quantity of charcoal, graphite and diamond present in a mixture.\* The finely powdered substance is treated by the method described for the preparation of graphitic acid; care, however, must be taken that not more than five grams of the mixture are used at once, as otherwise explosions may take place. In order to separate the diamond from the graphitic acid, the residue is gently ignited and again treated with the oxidizing mixture. The process is repeated until the whole of the graphitic acid has disappeared, but any diamonds which may be present remain unaltered.

According to Luzi the simplest chemical formula for graphitic acid is  $\rm C_{24}$   $\rm H_9$   $\rm O_{13}$  and according to Berthelot  $\rm C_{28}$   $\rm H_{10}$   $\rm O_{15}$ .

Stingle, who made a series of investigations relative to the existence of "amorphous" and "crystalline" graphite in nature as first recognized by Brodie, arrives at the following results:— Graphitic acid, produced according to the methods of Brodie and Gottschalk, from an earthy, previously cleaned Bohemian graphite, does not show any crystal leaves or scales, but represents a yellow amorphous powder of extremely fine division. graphitic acid, however, produced from graphite from Ceylon and crystalline varieties, exhibits under the microscope fine lamellar crystals. If, further, graphitic acid produced from Bohemian graphite is decomposed by heat, the resulting black product (pyrographitic acid) has a high colouring and covering quality, and resembles in this respect and may be said to be identical with ordinary lampblack, while the black decomposed mass resulting from graphitic acid, produced from crystalline Ceylon graphite, has little colouring or covering quality. Stingle uses these two qualities, colouring and covering power, to divide the graphites in practice into "amorphous," possessing these qualities and "scaly or crystalline," devoid of these. in 1869, and other noted chemists pointed out the chemical and physica' differences of the graphitic acid produced from the different varieties of graphite, the character of the latter depending entirely upon the physical aspect and probably upon the molecular structure of the graphites employed. They confirmed further that the pyrographitic acid, (the black mass resulting from decomposition of graphitic acid through heat) produced

<sup>\*</sup> Ann. Chimie et Phys. XIX, 399.

from Bohemian or amorphous graphite possesses a high degree of colouring and covering quality, while pyrographitic acid from crystalline or Ceylon graphite does not possess this quality.

From the foregoing it appears that there are in nature several modifications of graphite, and on this point Staudemeyer, who made a special study of the transition of pure carbon to graphite at higher temperatures says, that according to the present knowledge and development of the chemical-physical nature of the graphites, the formation of the two distinct varieties "amorphous" and "crystalline" graphites out of free carbon under certain conditions can now hardly be doubted and therefore he holds, that there is in nature a series of modifications of graphite carbon.

#### CHAPTER II.

# OCCURRENCE OF GRAPHITE.

# GENERAL FEATURES OF GRAPHITE DEPOSITS.

Deposits of graphite are widely distributed over the globe, but so many of them afford a mineral of an impure and undesirable character, and many again are so remote from transportation facilities, that only a few are of such a character as to be of commercial importance. All over the United States and Canada. graphite occurrences can be counted by the hundreds, but so far a very small percentage warrant exploitation. Experience has demonstrated that actual mining should be undertaken with great precaution and with a broad knowledge of the characteristics of graphite ore and of the exigencies of the market. some instances the deposits have contained a sufficient quantity of graphite, but it was so intimately mixed with other minerals such as quartz and mica, that it was impossible to separate the graphite at a sufficiently low cost to enter commercially into competition with graphite obtained from other deposits. The graphite from such deposits has been proved in the laboratory to be of a good quality; but commercial processes have not yet been devised for its profitable separation, especially from mica.

As outlined in the preceding paragraph natural graphite is divided into two classes, viz.: crystalline and amorphous, the former representing the graphite which has a "lamellar," "scaly" or "flaky" structure and which in itself is a nearly pure carbon, although it may sometimes occur in very fine, minute flakes, while all the other forms of whatever occurrences and character are referred to the "amorphous" class. The "scaly" or "crystalline" variety represents the graphite, that can be used for all purposes, for which this mineral is required; but on account of its limited occurrence, compared with that of the amorphous variety, it is used mostly for the finer purposes of manufacture such as refractory products, crucibles, lubricants, electrotypes and pencils, for which purposes it is specially adapted on account of its freedom from all impurities. The "amorphous" variety.

on the other hand, occurs much more abundantly in nature, but on account of the great difficulty experienced in separating the mineral from the gangue, a great many of the deposits containing amorphous graphite are at present of no commercial value and will remain so, until a cheap process for the extraction of the mineral is found. On account of this difficulty of purification the amorphous graphite is not used for the finer purposes of manufacture as lubricants or for the better classes of pencils, for electrotypes, etc., save in a few instances, as in some of the Bohemian occurrences, where a very pure product is obtained.

# Graphite Associated with Precious Metals and Iron.

Graphite is sometimes found in mineral veins or rocks associated with precious metals; it is even at times mistaken for a metalliferous ore. In the Sunnyside extension mine, San Juan, free gold is found associated with graphite in quartz.\* Graphite occurs also in some quantities and seems to be connected in some way with the occurrence of silver. In the once famous Silver Islet mine, † north shore of lake Superior, in most of the ore, pieces of trap and graphite can be noticed in pink spar, while from the graphite start out dentrites of silver. It is reported, that in this mine the silver was invariably associated with graphite. but that the latter occasionally occurred alone in small bunches and pockets without the presence of silver. The association of graphite and the occurrence of other forms of carbon in connection with metalliferous veins, can be noticed in many localities of the lake Superior Region (Ont.), and the favour, with which it was regarded as an indication, arises from its close association with the rich ore of Silver Islet. The anthracite-resembling form, which is occasionally found in the veins, as well as in the enclosing rocks, would seem in composition to be almost, pure carbon, showing as it does, no volatile matter on being heated. It has been described as altered bitumen in the "Geology of Canada, 1863."

Graphite is, occasionally, intimately mixed with magnetic and hematitic iron ore, both in Scandinavia and in Canada,—specimens of the large magnetic ore bed of Hull near Ottawa show sometimes alternate layers of coarsely granular magnetite and of red hematite, the two being somewhat intermingled at

<sup>\*</sup> Mines and Minerals, 1901-1902, page 515.

<sup>†</sup> Geology of Canada, 1887-88, Part II, page 28. H.

the junction. Grains of greenish feldspar are disseminated in the magnetite, and both it and the hematite contain imbedded crystalline plates of graphite, a tenth of an inch or more thick. A film of scaly graphite, moreover, coats the free surface of the hematite layer. The hematite of the Woodstock mines, New Brunswick, is reported to be often seamed with thin layers of graphite.

According to Fowler\* graphite is found disseminated in the magnetic iron ore in Franklin, New Jersey, and at one time was an obstacle to the working of iron in the Catalan forge. Beck has also described as occurring near the Natural Bridge, in Lewis county, New York, a mixture of chlorité, graphite and red iron ore, the latter amounting to about one half of the mass.†

The presence of graphite has been noticed in some of the most famous meteorites, as follows: Lenarto, Ungary (1815), near Benedego in Bahia (1816), Bohumilitz in Bohemia (1829), Sevier in Crosby Creek (1840), the latter containing large lumps of graphite; Congford in Tennessee (1845), Chartago in Tennessee (1846), Seelasgen in Brandenburg, Germany (1847), Chesterville in South Carolina (1849) and Kaba in Ungary (1857). All these meteorites are of great scientific interest, inasmuch as they have yielded most of the very fine graphite crystals.

# Classification of Deposits.

Far more important from an economic point of view are those occurrences which comprise large workable deposits, and which are the subject of exploitation in many parts of the world.

The bulk of the world's supply of graphite is chiefly derived from Ceylon, Austria, Bavaria, Siberia, England and the United States; workable deposits, some of them of large dimensions are also being mined in Canada, Italy, and recently in Mexico, while discoveries of deposits are reported from Western Australia, New South Wales and New Zealand.

The mode of occurrence of graphite shows great variety, affording quite a study in itself. Although the technical importance of the mineral has been recognized for a great many years even for over a century, and although the study of the various occurrences has been facilitated by increased mining develop-

<sup>\*</sup> Rogers, Final Rep. Geol., New Jersey, page 64.

<sup>†</sup> Mineralogy of New York, page 26.



Outcrop of Graphite on Lot 14x, range 10, Township of Buckingham. Property of the Diamond Graphite Co. Note the Laminated Structure (X).

ment, our knowledge regarding the geological position and origin of the mineral is still imperfect.

Graphite is found in the oldest crystalline formations, distinguished from all the other younger rocks by their absence of any organic matter. But it is also found to some extent in the sedimentary strata, very often in connection with coal, forming a kind of graphitic anthracite, in which form it is mined to some extent in Rhode Island. Similar graphitic carbon is also found in the coal basin of Massachusetts and also in connection with a deposit of anthracite in southern New Brunswick. In the northern Alps near Paltenberg graphite is found associated with coal seams in the coal formation, which is composed of schists, limestone and conglomerate; very often the graphite obtained from this locality has the lustre and appearance of anthracite, and is distinguishable with difficulty from the latter. It is also found in a number of places, in association with beds of altered clav slates and shales of more recent date than the crystalline rocks. ally speaking, the largest and most valuable deposits appear in all cases to belong to the older crystalline formation of pre-Cambrian age. In Canada the economic deposits occur in gneiss and crystalline limestone of the Laurentian formation, in that part, designated as the upper portion of the Grenville series, which is cut by a great many dikes and masses of eruptive rocks. In the State of New York the workable deposits of Ticonderoga occur in a formation similar to that found in the northern part of Ottawa county. The graphite of Sturbridge, Massachusetts, forms bedded veins in a foliated gneiss, which have been worked to a depth of 60 and 70 feet. The same may be said of the graphite occurrences near Brimfield and North Brookfield in the same state and of other localities in Connecticut and Vermont. The large graphite deposits of Ceylon, which have been worked since 1827 and which appear to be inexhaustible, occur in granite rocks, belonging to the Laurentian formation. The deposits in Bavaria and Bohemia, which have been worked for several hundred years, are exclusively confined to the gneiss formation, while the Siberian deposits near Irkutsk, occur between syenite and granite.

All graphite deposits may be classed genetically into 3 groups,

- 1. Veinlike occurrences.
- 2. Bedlike occurrences.
- 3. Disseminations through the country rock.

#### Veins or Veinlike Occurrences.

In the first mode of occurrence, that of veins, the graphite constitutes the filing of veins and cracks in gneiss, crystalline limestone, pegmatite and granular eruptive rocks. Both hanging and footwall are easily discernable, and the direction of the vein is independent of the strike of the rocks traversed. Sometimes branches and narrow apophyses run out from the large graphite veins between the layers of country rock; they dwindle away and along their line of continuation isolated plates, patches and pockets of graphite are deposited.\* Fissure veins of this character occur in the country north of Ottawa and near Grenville, county of Argenteuil. Fig. 4.



Fig. 4.—Fissure Vein of Graphite, Buchingham, Grenville, Can.

The deposits of Ceylon, Borrowdale (Cumberland, England) and Batugal (province of Irkutsk, Siberia) all belong to this class.

In Ceylon true fissure veins of a scaly and fibrous graphite intersect granitic or closely associated rocks, which are sometimes highly decomposed and consist then mainly of kaolin and similar decomposition products. While the geological horizon of the

<sup>\*</sup> The mineral contents of the majority of fissure veins is very simple, in by far the larger number of cases the graphite itself filling the veins; it very often consists of parallel fibrous or rod like aggregations, the direction of the fibres being vertical to the vein walls.

containing rocks is not definitely recognized, it appears, that they represent some portion of the Archæan. In Borrowdale (Cumberland, England) a fine scaly graphite occurs in veins, which traverse a greenstone porphyry. The gangue is chiefly calcspar and quartz, in which occur nests and lumps of a very fine graphite, especially suitable for the manufacture of pencils.

In Batugal, province of Irkutsk, Siberia, a finely fibrous graphite, purer than that of Borrowdale, occurs in veins, which run through a granite or dioritic rock, while in the closely adjoining limestone, which is altered by contact metamorphism, are great lumps of pure graphite, suitable for the manufacture of pencils.

#### Bedded Veins or Masses.

As to the second form, that of "bedded veins or masses," it must be said, that this mode is very often met with, and that large deposits of this character have been discovered and sometimes profitably mined in many parts of the world.

The principal feature of bedded veins is that their general outline and main direction conforms with the stratification of the country rock; they form in the majority of cases disconnected layers, lenticular masses or chain-like accumulations between the layers of the enclosing formation, giving off sometimes branches, which again are accompanied by parallel lenticular masses or numerous disseminations of graphite through the country rock.

This form of occurrence can also be noticed in a number of localities in Canada, especially in the townships of Buckingham, Grenville, and also near White Fish lake, in Ontario.

Near Passau, on the Danube, Bavaria, gneissose rocks occur impregnated with scaly graphite which at times appears as lenticular masses of rich mineral. Fig. 5.

Here they are found chiefly in the immediate neighbourhood of intercalated beds of granular limestone, altered by contact metamorphism. Both the graphite bearing rock and its near neighbours are much decomposed, so that kaolin and other decomposition products are found in intimate association with graphite deposits. The genetic connection of these rocks with the Ceylon type is very close. The lenticular form of the deposits as found in Schwarzbach-Krumau (Bohemia), their geological relationship with intercalated limestones, their frequent association with kaolin and other decomposition products, connect them closely with the

Passau type, from which they are differentiated by the more compact, less crystalline character of the graphite.

On the northern border of the central zone of the Styrian Alps, near Kaisersberg, is a highly metamorphosed system of carboniferous shales, clay slates, limestones and conglomerates with bedded

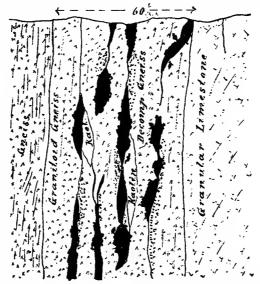


Fig. 5.—Lenticular Masses of Graphite near Passau, Bavaria.

marses of coal and coal seams, the coal of which has passed into graphite, which preserves completely in some cases the appearance of coal, from which it is derived. It is very compact, very pure, and often extremely hard.

## Disseminated Graphite.

As to the third class of deposits, that is the disseminated varvariety, this mode of occurrence is less desirable from a mining point of view than the other two, as described above, since it involves the handling of a very large quantity of rock, depending upon the percentage of graphite contained therein, in order to obtain the pure article. However, it must be mentioned, that most of the successful mines on the North American continent have been working on deposits of this character, although the vein occurrences as a rule are of too limited an extent to render their commercial extraction lasting and profitable.

This disseminated variety occurs as scattered scales or plates

through certain portions of the gneiss, limestone or other similar strata; it occurs often in proximity to veins, and in these cases it is supposed that the graphite has thence been distributed along the planes and fissures of the rock in contact from the veins itself. This has been more particularly observed, where the adjoining rock is a mica gneiss, but similar occurrences are also found in limestone and other rocks belonging to the older series.

In the majority of cases disseminated graphite occurs in the neighbourhood of eruptive rocks, like granite, diorite, porphyry or other intrusive rocks, in fact, some of the richest portions of disseminated graphite have been found near the contact of the country rock with these rocks, as observed in some localities near Buckingham. Fig. 6. It can therefore be reasonably inferred that the

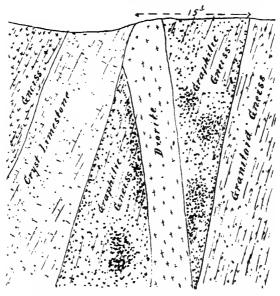


Fig. 6-Disseminated Graphite, Buckingham, Que.

presence of these intrusive dikes has had a beneficial influence upon the formation of the deposits in the adjacent strata.

Mica schists contain disseminated graphite to such an extent, that they become graphite mica schist, as in some parts of Saxony, Germany, and in the Pyrenees; some of these occurrences have been mined profitably, the product obtained, after freeing it from grit, being highly suitable for lubricating purposes.

# Occurrence of Graphite in Canada.

Deposits of graphite are widely distributed over the eastern portion of Canada, but in many localities they are so limited in extent, or are so impure, that their exploitation is unprofitable. Most of the workable deposits are of the disseminated class, and while it is true that fissure veins occur abundantly throughout the Laurentian formation, it must be stated that the best returns have always been obtained from those portions of the formation, where the mineral is found disseminated in the form of fine scales or flakes, and sometimes accompanied by irregular bunches or pockets of amorphous graphite.

Though the vein form frequently occurs at most of the points, where attempts to work the graphite have been made and has shown in such cases a mineral of great purity, the uncertainty of such deposits is so great, that the employment of capital on a large scale in this mode of occurrence would scarcely be warranted.

The most important of the graphite deposits are situated in the townships of Buckingham and Lochaber, Ottawa county, in the townships of Grenville, Argenteuil county, province of Quebec, in the counties of Lanark, Leeds, Frontenac and Addington, province of Ontario; in the vicinity of St. John, and at Kings and Westmoreland counties, in the province of New Brunswick; in the province of Nova Scotia, and at Alkow Harbour, Dean Canal, British Columbia.

# Province of Quebec.

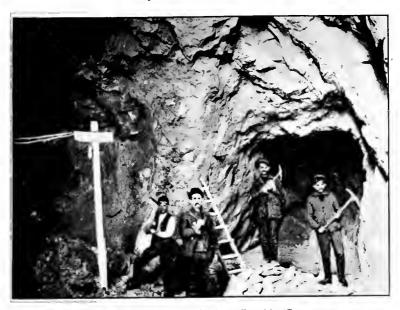
Mining for graphite in Canada dates back to the year 1847, when, according to Sir William Logan, "Mr. Harwood, of Vaudreuil, took out several tons of graphite from a mineral bearing vein in the crystalline limestone near Grenville." We find further reference to graphite deposits in the reports of the Geological Survey for 1863, by Sterry Hunt, whose treatise on this subject may be summarized as follows:—

1. "Plumbago occurs in the altered rocks of the base of the palæozoic series in the Eastern townships, generally finely disseminated in calcareous or argillaceous shales, rendering them soft, unctuous, black and shining; but nowhere does it occur in sufficient quantity to be of economic value. Examples of these plumbaginous slates are to be seen in Granby, Melbourne, and St. Henry, in

### PLATE VI.



The Milling Plant of the Buckingham Graphite Co., near Buckingham, P.Q.
This Mill has been entirely Reconstructed and Overhauled
by Mr. Brummel, M. E.



Western Drift, Buckingham Graphite Co.

the latter place enclosing graptolites. The altered Devonian limestones of Owl's Head are also plumbaginous."

- 2. "Workable deposits, however, occur only in the Laurentian series, in the townships of Burgess, Lochaber, and Grenville, in beds or seams of from a few inches to two or three feet in thickness. These deposits occur generally in limestone or in their immediate vicinity, and granular varieties of this rock often contain large crystalline plates of plumbago. They are often interrupted, giving rise to lenticular masses, which are sometimes nearly pure and at other times mingled with carbonate of lime, pyroxene and foreign minerals. At other times this mineral is so finely disseminated as to give a bluish gray colour to the limestone and the distribution of bands thus coloured serves to mark the stratification of the rock."
- 3. "The plumbago of the Laurentian series is, however, not confined to the limestones. Large crystalline scales of it are occasionally disseminated in pyroxene rock, in pyrallolite and sometimes in quartzite and feldspathic rocks, or even in magnetic oxide of iron as in the Hull ore bed."

The above represents the gist of conclusions reached by this noted geologist and may still be applied to-day to the graphite deposits of the Quebec region. Since that time the deposits in Ottawa county have been opened up and studied, and in the following, additional information regarding the characteristics of the ore bodies will be given.

The economic useful deposits of graphite are practically confined to the crystalline limestones and associated gneisses, which form the upper members of the so-called Grenville series, formerly regarded as representing the middle or upper part of the Laurentian formation. These rocks are cut frequently by intrusive dikes of granite, diabase, pyroxene, etc., and the graphite occurs in the form of disseminated flakes or scales, in gneiss and limestone or as true veins, both columnar and foliated, which, however, are also found frequently cutting the granite and other igneous rocks as well as the gneiss. The workable disseminated graphite is, however, in the majority of cases confined to gneiss. A good illustration of this is the Walker mine, on lot 19, range 8, township of Buckingham. The workable graphite occurs here in a tunnel, and is confined to bands of a grayish gneiss, a "sillimanite," while a band of limestone setting through the latter contains nothing of importance. These bands of gneiss have a thickness of from one to

thirty feet, and carry from nothing to sometimes forty per cent. of graphite. The gneiss is often found in a rusty condition, especially in the vicinity of intrusive dikes of granite, pyroxene and diabase, which apparently have developed the formation of iron pyrites in the adjacent rock.

Prof. Osann, of Germany, made a thorough examination of the Walker graphite locations in the year 1899, and, as his report contains some points of interest as to the occurrence of the mineral, an abstract of the same is given in the following:—

"The workings of the Walker Mining Company comprise lots 19, 20, 21 and 22, of range 7 and 8, township of Buckingham. There are a great number of openings all over this area, but only a few of them could be examined. Of special interest are the following points:—

1. "The graphite occurs as the filling of veins and cracks in the gneiss, granular limestone, pegmatite and granular eruptive rock. Fig. 7. The direction of these veins is independent of the

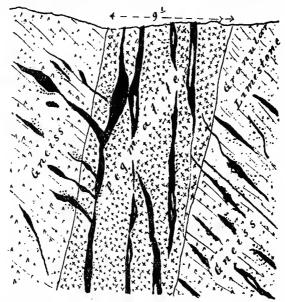


Fig. 7.—Graphite Veins occurring on the property of the Walker Mine.

strike of the rocks traversed. Thus in one place the gneiss, having a s rike of N.E. 70°, with relatively flat dip, is cut by four graphite veins, all parallel and about two inches thick, striking N.W. 20°, with almost perpendicular dip. Near Nellys pit, there is a pegma-



Outerop of Graphite on Lot 14B, Range 10, Township of Buckingham. Property of the Diamond Graphite Co. Note the Schistose Structure (X).



tite vein in the gneiss from six to nine feet wide; in this in a relatively small space there are several graphite veins of several inches in width. In the pit itself the veins are collected on the boundaries of the granular limestone, the gneiss and plutonic rock, the latter forming a dike. The thickness of these veins is from six to eight inches. From the material on the dump, it can be noticed how narrow branches have run out from the main graphite veins between the layers of gneiss, accompanied by small bunches or pockets of graphite; the granular limestone is strongly impregnated with graphite from the veins. One gets the impression that the loose structure would make the penetration of foreign substance particularly easy. In the main pit, in a horizontal tunnel, where granular limestone and gneiss is developed, no graphite veins could be noticed; but both rocks were abundantly impregnated with the mineral in scales and plates. An opening, made about fifty paces above this working in the same hillside, shows a graphite vein of several inches in thickness in an altered gneiss and granular limestone. The complete similarity of these relations show that the graphite is here a typical vein material, but the veins themselves are younger than the pegmatite, and therefore certainly younger than the gneiss and granular limestone cut by the pegmatite.

- 2. "The vein filling consists of graphite in by far the larger number of cases; it then is composed of parallel fibres or columnar aggregates, the fibres being vertical to the walls of the vein, as is very common in a number of localities in Ceylon for example. In several cases green apatite and scapolite occur with the graphite. The occurrence of apatite appears to be not uncommon, and reminds one of the occurrences of the same mineral in the graphite veins of Ceylon. Grünling describes the occurrence of apatite in graphite in Ceylon in the form of large crystals along with iron, magnesia, mica, calcite, quartz and pyrite. Coarsely crystalline calcite also plays an important role in the Ceylon veins.
- 3. "The occurrence of graphite is connected with the appearance of massive eruptive rocks, which in mineralogical composition are very similar to those occurring in connection with the apatite occurrences. It appears that the occurrence of the graphite is connected with the contact of these eruptive rocks with gneiss and granular limestone. The latter is in places very much altered; there has been especially a large production of scapolite, pyroxene and titanite. Such altered limestones are so like the pyroxene rock from the neighbourhood of the apatite veins as to be mistaken for it.

"Very frequently mica is met with in connection with graphite and the parallel arrangement of this mineral develops a parallel structure of the rock. The graphite occurs generally in the portions rich in mica and forms irregularly bounded ragged masses, usually elongated in the direction of the schistosity, and appears to be closely associated with iron pyrites. The same association is found everywhere in the limestone rich in graphite, which is here mined for this mineral. Very often the graphite lies in the form of thin lamellæ in the cleavage of the mica, suggesting primary intergrowth and surrounds it upon the edges. Again it is seen along transverse cracks and fissures in the mineral, particularly the spaces, caused by the mica crystals opening up along the cleavage lines under mechanical stress, are filled by it. Portions poor in mica are avoided by the graphite, but it is sometimes seen here and there invariably on the edges of various feldspar grains in long narrow shreds; it follows all the bends and curves of the outline. This whole mode of occurrence of the mica shows that it is the youngest of all the constituents, and formed in the rock after its solidification, that is the product of infiltration."

It may be of interest to summarize in the following the results arrived at from a closer study of the graphite occurrences in the Buckingham district:

- 1. The rocks of the Buckingham district are largely composed of gneiss, crystalline limestone and quartzite, which are cut by numerous masses of eruptive rocks like pyroxene, granite and also, but not so frequently, by dikes of diorite and diabase. The general trend of these rocks is north east, varying of course at different localities, which is likely due to large intrusions of eruptive rocks, as seen on the Lievre river.
  - 2. The graphite occurs in these rocks:
    - (a) In columnar or foliated variety in true fissure veins cutting gneiss or the different eruptive rocks.
    - (b) As the flaky or amorphous variety in bedded lenticular masses in crystalline limestone on or near the contact of the latter with eruptive rocks; such as granite, pyroxene and less frequently diorite and diabase.
    - (c) In the form of disseminated flakes in limestone and gneiss; less frequently in quartzite, pyroxene or associated with iron ore as in the Hull iron ore deposits.

# THE GRAPHITE MINES AND MILLS IN THE PROVINCE OF QUEBEC.

The early history of graphite mining in the Buckingham district dates back to about forty years ago. The first mill of any importance appears to have been erected by the Lochaber Plumbago Company on the Blanch river, lot, 28, range 10, township of Lochaber. The mill was run by water power and was supplied with a battery of eight stamps and two circular buddles. The ore was stamped in water and then afterwards passed over the buddles and then through buhr stones and screens. It was obtained from several points in the vicinity and principally from lot 24, range 8, and lots 23 and 24, range 11.

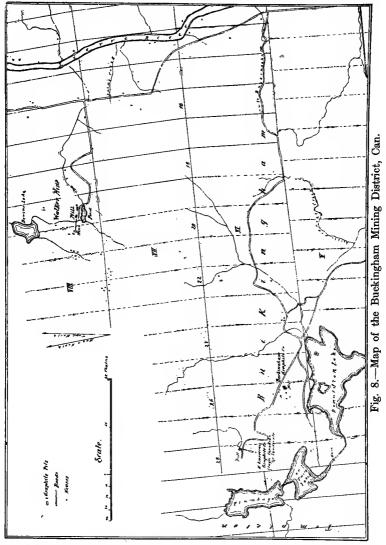
Two other mills were erected in the early days of the industry; one was at the Garret lot (on McNaughton creek) about two miles east of Buckingham and the third one on Fernie creek, which is a discharge from the Twin lakes. The last was burned down and the other was abandoned and gradually fell to pieces. These old mills have been supplanted by three new ones, which now have been in operation at intervals for some years.

The companies working at present in the Buckingham district are the Anglo-Canadian Graphite Syndicate (at present in liquidation, which took over the property of the North American Graphite Co.) the Buckingham Company and the Walker Mines, the latter being operated now by Mr. H. P. H. Brumell.

The Anglo-Canadian Graphite Syndicate: (in liquidatian)— The works of this Company are situated near the north-east corner of lot 28, range6 of the township of Buckingham, see Fig. 8, at a distance of eight miles from Buckingham, where there is a station on the Canadian Pacific Railway. The plant comprises a mill, a dryer and engine house, storehouses, repair shops and a comfortable boarding house. Water for the supply of the boilers and for domestic purposes is obtained from a creek and a small pond to the west of the mill building. The whole plant is lighted throughout with electricity.

The mine is close to the mill, with which it is connected by a tramway. For about one hundred feet it represents an open cut, continuing then into a tunnel in the side of a hill for several hundred feet, exhibiting an irregular opening with occasional vent

holes in the side of the roof to the open air. The main direction of this open tunnel is north east 10°. The rock, in which the tunnel is run, is for the most part a bluish gray quartz, containing small



particles of iron pyrites, which, on decomposition, gives a rusty appearance to the enclosing rock. The graphite is found here in the disseminated form, representing a sillimanite gneiss. Another opening, No. 2, located to the west of the tunnel, is 110 feet long.

At the southern end two drifts have been run into the eastern The graphite here also belongs to the disseminated class and occurs in a highly quartzose rock. The ore appears to be richest in certain streaks, which show no definite lines of demarcation from the surrounding rock, but generally a transition can be observed from rich to poor and from poor to barren rock. Sometimes the richest of the ore passes suddenly into the rock that is quite barren. About six hundred feet to the south of No. 2, another exposure, No. 3, of disseminated ore, is noticed in a band from two to three feet wide running N.E. 35° with a steep dip to the south. To the west of this pit well banded gneiss appears overlaid by a band of disseminated graphite. Amongst the many openings which can be seen all over the property, the one which occurs on the brow of a hill No. 4, at a distance of about half a mile from the mill in a southern direction, is the most important. This is an open cut towards the north, which follows a deposit of graphite measuring at the surface only a few inches, but widening out, it is said, at a depth of twelve to fifteen feet. The ores are of the disseminated class. At the mouth of the drift, leading to the pit, a shaft was sunk fifty feet deep, in which ore was said to be found all the way down. At the north-west corner of this pit the rocks form an anticline, the dip showing to the east and west on either side of the axis of the fold. Owing to this structure the graphite band appears as a sort of blanket overlying a boss of massive quartzose rock similar to that found in pit No. 1. Gneiss, containing mica and graphite weathered and rusty, overlies the ore. The general run of the opening is N.E. 10° with a dip of 45° towards the east, and this may be taken as representing the strike and dip of the deposit worked.

The mill process used is dry with a wet treatment for the tailings by means of Brumell's patent wet boxes. There are six Hooper air jigs, four buhr stones, two sets of rolls and two jaw crushers with all accessories in commission. A hundred h.p. slow speed engine, fed by two horizontal boilers of 100 h.p. each, drives the mill. There are altogether ten grades produced, from a very fine crucible and lubricating graphite, down to the cheaper grades. The capacity of the mill is about five tons of finished graphite per week.

The Buckingham Company.—This property formerly known as the Pugh and Wearts Mine comprises lots 26 and 27 in range 6 of the township of Buckingham. The mill is situated on lot

26 near the south-eastern corner, all the excavations and pits being located on lots 26 and 27. The distance is seven miles from the Canadian Pacific Railway station. Amongst the many openings all over this property only the following of importance may be mentioned. In pit No. 5 (see map), about a quarter of a mile from the main road, a graphite vein, showing on the surface a thickness of eighteen inches, was followed down to a depth of seventy feet, where the vein was found to split up into several stringers of only a few inches wide. The strike of this vein is N.W. 85° with a dip of 75° to the south. The north wall consists of massive granite, while the exposures to the north and east of the pit were found to consist of a rusty weathered gneiss. The walls of the vein exhibit a thin coating of calcite, and in the southern wall near the present working face a number of small apatite crystals can be noticed. From an examination of the dump, it appears that a light colored feldspar is the principal gangue rock. The pit measures sixty feet long, from three to fifteen feet wide, and its total depth is reported to be seventy feet. Pit No. 6 is the one on which most of the work had been done; it is located on the side of the slope to the south west of the mill. The opening is eighty-five leet fong with a main trend of N.E. 40°. The southeast side is composed of coarse granite with a large amount of feldspar. The occurrence of graphite, which is mostly of the disseminated variety, is very irregular, showing small veins of graphite or bands of gneiss sprinkled with flake graphite, from several inches up to four feet in thickness. A graphite bearing zone can be noticed in the north-west side of the opening, where it is underlain by the granite and overlain by a rusty gneiss. the immediate vicinity of this pit towards the east, a shaft has been sunk to a depth of thirty-five feet, which shows a continuation of the graphite zone in the direction of the mill. south west of Pit No. 6 on the southern slope of the same hill towards Donaldson lake is another opening, No. 7, measuring about sixty feet long and from twenty-five to forty feet deep. The disseminated ore occurs in a band from two to three feet wide dipping steeply towards the west. The exposed surface of the ore is decomposed, forming a red sandy deposit.

The process by which the ore was refined in the mill several years ago was dry throughout; the ore was first roasted to drive off the moisture and sulphur and to make the rock easier to crush. It passed then through a system of rolls and bolts.

# PLATE VIII.



Big Outcrop of Graphite at the Extreme North End of Lot 14B, Township of Buckingham.

The Buckingham Graphite Company.—The general manager of this Company is Mr. H.P.H. Brumell, M.E., the well known graphite expert. The property comprises 1,388 acres of graphite lands, the largest area owned by any one graphite company in Canada. There are a great many openings and pits all over the property especially on lot 20, range 8, Buckingham, while the milling plant is located on lot 19 B, range 8, in the valley of a small stream, which gives a constant supply of good water for the boilers and domestic uses. Pit No. 1, Fig. 8, from whence the bulk of the ore is drawn, lies to the west of the mill and is connected with the latter by a tramway 1,100 feet long. A tunnel has been run into the side of the hill for a distance of 150 feet. At this distance the tunnel widens out to ten feet, while the height is from twenty to thirty feet. The ore is a rich disseminated graphite and occurs in gneiss. There are also limestone bands present, which carry also the mineral in a disseminated form, but it appears that the workable portion of the deposit is confined to Small patches of iron pyrite are occasionally found in the latter. Along the slope of the hill to the north-east and to the south-west, a number of prospecting pits also show the disseminated mineral in this part of the formation in such a quantity as to render the property, if properly developed, a valuable one. limestone at the tunnel is an interstratified bed and contains numerous intrusions of gray gneiss through the calcareous mass. The strike of the graphite bearing gneiss is N.E. 50° with a dip to the north-west of 75°. There are about thirty openings all over the property, showing in a more or less degree the abundant presence of the mineral, and a large amount of money has been spent in development work. Recently a new pit has been opened. about 100 yards to the north-east of the big pit; this new pit according to the latest reports is showing up very well affording a very high percentage of large flakes. The mill building is 122 by 72 feet and contains all the necessary machinery for crushing, separating and cleaning of the mineral for the market. The mill is operated under the so-called "Brumell Process," and the production is two tons of finished graphite per day.

Another property which has come lately into prominence is that owned by the Diamond Graphite Co., composed of New York capitalists. The credit of establishing the value of this occurrence is due to Messrs. J. J. Tonkin, T. T. Hazlewood, J. S. DuBois and C. L. DuBois, all officers of the above company, who

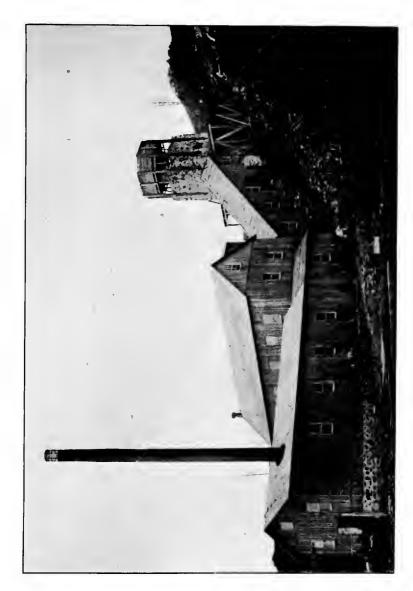
spared neither effort nor money to find out the exact nature of the occurrences, which to some extent were concealed by drift and overlying country rock.

The property comprises lots 14 A and B of the 10th range of the township of Buckingham, Labelle county, and covers an area of about 200 acres. It is splendidly adapted for mining purposes, the graphite occurring on the slope of a hill extending over the rocky ridges in an east-westerly direction.

A great many openings have been made all over the property, while 14 drill holes to a depth of 14 feet have aided in locating new ore bodies and in forming an idea of the character and extent of those deposits tested by shallow openings. It has thus been illustrated that so far the productive graphite area covers approximately over half a million square feet and has been thoroughly tested by more than 50 prospecting ditches, cross-cuts, pits, excavations and drill holes. Outside of this well prospected area, many indications of the mineral have been found in close proximity, but little or no work had been done on them.

The graphite is exclusively of the flaky variety and occurs in a disseminated form through the highly decomposed matrix of a rusty, weathering, dark greenish gray, sometimes greenish black schistose rock with occasional narrow bands of a quartzose Sometimes solid bodies of the latter underlie the graphitic rock comformably. Frequently the latter is traversed by dikes of a pure feldspar, quartz, hornblende granite and mica diorite and in the proximity of the latter sometimes pure vein graphite is found. The graphite rock, as can be noticed in most of the openings, is highly charged with iron pyrites, which on the more exposed portions is decomposed and imparts then to the rock matrix a whitish tarnish. Its foliated character is well pronounced and the deposition of the larger graphite flakes seems to be more abundant between the foliage of the rock. A series of tests with a number of average samples of the graphitic rock have shown that the latter contains approximately over ten per cent. flakes.

At the time of this writing a mill for the treatment of the ore is under construction at the foot of the western extremity of the high ridge. The process adopted is that of the International Dry Concentrator Company of 42 Broadway, New York and will be dry throughout. The ore will be carried by means of an inclined tramway from the various openings on the hill to the



[New Graphite Mill of the Diamond Graphite Co., at Buckingham.

stationary calcining furnace, where the sulphur is carried off. It then passes through a series of crushing machinery, as jaw breakers and rolls and after screening is delivered to the Krom Air Jigs, where the separation of the flakes from the gangue is effected. After screening and polishing the flakes the product is ready for the market. It is intended to manufacture only the higher grades as crucible and lubricating stock.

The mill will have a capacity of 100 tons of rock per day in two shifts, the output to be about 8 tons of the finished article from above quantity. The mill building proper measures 26 by 72 feet; additional buildings are provided for the reception of the boilers, power machinery, etc. The mill will be operated by electricity, developed by steam and later derived and transmitted from a water power at Masson village, on the Lievre river, at a distance of 9 miles. It will be divided into several sections, each section to be operated by a special motor. The boarding house measures 20 by 56 feet, is two stories high and will accommodate 40 men.

The Dickson Graphite Locations.—A great many indications of graphite point to the existence of a mineral range in the fourth concession of the township of Buckingham.

On lots 1, 2, 3, 4, 5 of that concession a number of openings have been made especially on No. 3 and 4. On lot No. 3 a fine showing of graphite has been stripped for a length of 40 feet and a width of 30 feet. The ore in this stripping is mostly of the disseminated variety, small veins of foliated graphite may also be seen at intervals. In immediate vicinity to this occurrence is another outcrop, 37 feet long and 9 feet wide. About two chains further east across the strike more ore is exposed, and a number of shallow test pits exhibit graphite of the flaky variety. Walking north-eastward along the strike of the formation for a distance of 6 chains several test pits can be noticed, all showing more or less indications of graphite.

All these outcrops evidently indicate the continuance of the graphite bearing formation to the north.

On lot 4, concession 4, about 15 chains north of the southern end and 10 chains from the eastern side of this property a stripping of 100 feet long and 8 feet wide can be noticed. It apparently extends along the strike of the formation and shows ore throughout its length and breadth. Parallel with this exposure and about 75 feet north-westward is another stripping 35 feet in length

by 9 feet in width, while 15 feet further west is still another opening 16 by 9 feet. Both these openings show considerable graphite and are presumably on the same ore body.

Some work has also been done on lot 28, concession 4, Lochaber township. About the centre of this property, which is known as the Pearson lot, an outcrop of good ore can be seen in a hillside stripping 18 feet long and 8 feet wide. The graphite occurs here in association with crystalline limestone in a disseminated form. Across the gully to the eastward is a similar side-hill exposure, 67 feet in length and showing ore throughout a width of 33 feet. Further stripping would undoubtedly show up a still greater width of mineralized zone. The strike is about northeast and south-west. For 250 feet along the strike to the northeast, graphite ore occurs at intervals and further exploration and development would undoubtedly show up considerable quantities of ore.

The H. E. Dickson Graphite properties in concession 8, Buckingham township, comprise lots 20, 21 (north half) and the whole of lots 22 to 27, in all about 1,600 acres, and adjoin the well known Walker Graphite Mines.

The rocks exposed on lot 22 consist mostly of the typical rusty gneisses of the region together with crystalline limestone diorites, etc.

In the south-east corner of this property a stripping 18 feet in length and four feet in width exhibits a fine showing of disseminated graphite. About 150 feet to the north eastward is a small test pit, showing indications of a disseminated ore while ten feet further north is a stripping 25 feet in length and 5 feet in width. The strike of the formation is north 25 degrees east, and the glaciated and grooved exposure shows plenty of disseminated graphite, with bands of the foliated variety. One of the latter is about 7 inches wide. Following the strike to the northward for about 500 feet, indications are seen at intervals, and near the northern end of the hill, which slopes down to a small creek, is a stripping 19 feet long and four feet wide on rusty gneiss. Its strike appears to be north 40 degrees east and the deposit consists of both disseminated ore and the foliated variety. On the east side of the opening diorite is exposed for some 20 feet, while across the valley of the brook and on the same strike is an exposure of a coarse gabbro showing considerable foliated and crystalline graphite. In the gulley and 100 feet to the westward some good exposures of a foliated graphite can be noticed. There is no doubt that the conditions on this lot are very encouraging and careful prospecting and stripping should disclose some good ore bodies. On lots 23, 24, 25, and 26 from east to west a number of indications of graphite can be noticed. At one place on lot 26 a test pit on a hillside 6 feet deep showed graphite disseminated through the rock.

On lot 27 in concession 8, two strippings on a hillside disclose outcrops of graphite of both the disseminated and foliated variety. One opening has a length of 14 feet and a width of 9 feet. In it the graphite is pretty well distributed while about 25 feet further along the strike another graphite exposure can be noticed. The lower stripping exposed similar rock and ore and was 9 feet long and 11 feet wide. As good laminated ore was exhibited between the two principal openings, that is, over a distance of 47 feet, it is evident that the body of ore here is of appreciable extent.

## The Grenville Deposits.

Sir William Logan described as early as 1845-46 some occurrences on lot 10, range 5, of Grenville, county of Argenteuil, where mining was carried on for some years. But later the property was abandoned for over twenty years, when it was worked again for a short time as the Miller Mine. In 1898 it was acquired by the Keystone Graphite Company of Wilkesbarre, Pa., and a considerable amount of development work was done. Since then operations were again suspended. In the report of the Geological Survey for 1876 reference is made to the above locality, and a brief description is given of the occurrences of the mineral. In 1899 work was also begun on lot No. 9, lying to the east, by the National Graphite Company, Scranton, Pa., the rocks on both lots being practically similar in character. This property has also been abandoned.

The area was thoroughly examined by Dr. Osann in 1899 and an abstract of his report is as follows:—

"The country rock is for the most part crystalline limestone, which is cut by granites and other intrusives. The graphite usually occurs irregularly at, or near, the contact of the limestone with granite or diabase dikes, both rocks being present in the openings, also in irregular vein forms which are massive rather than colum-

nar in character, ranging in thickness from fifteen inches to two feet. These are not solid but apparently sometimes in dike matter."

"Several openings have been made on the property. In the main pit the rocks are limestone with bands of rusty gneiss, which are traversed by a white granite dike and this in turn by a dike of light green diabase. The graphite occurs principally in two irregular veins, and also in the granite mass, and there is a small vein on the edge of the diabase. The veins are shattered and mixed with the whitish, sometimes reddish granite."

"This granitic-looking rock has the aspect of a vein in some respect rather than a true dike. It carries several minerals, including scapolite, hornblende, graphite, pyroxene, pyrite, apatite and others. South of the principal opening, where mining has been carried on, the surface rocks for some distance appear to be all limestone, and in several small prospecting pits, sunk in this rock, a small percentage of disseminated flake graphite was observed."

The only company operating in the Grenville district at present is the "Calumet Mining and Milling Graphite Company," which owns altogether 258 acres of graphite lands on range 2 and 3 (on lot 16, range 2 and on lot 16, range 3) in the rear of Calumet station on the Canadian Pacific Railway. This property is situated 60 miles west from Montreal and the same distance east from Ottawa. The largest opening is located on the slope of a hill and about 150 feet above the railroad; it consists of an open cut into the hill at the end of which a shaft has been sunk to a depth of fifty feet. The country rock is gneiss, striking N.E. 70°, occasionally interrupted by bands of granite, quartz and crystalline limestone. There can be noticed, in the open cut and shaft, four veins of graphite from a few inches up to eighteen inches wide, which are reported to increase at the bottom of the shaft (which could not be examined on account of being filled with water) to a width of thirty inches. Sixty-five tons of this vein graphite were mined and sent to the Globe Refining Company of Jersey City, N.J., and yielded thirty-two tons of clean crucible graphite. The Morgan Crucible Company of London and also J. H. Gauthier and Company, Jersey City, used some of this graphite in their crucibles and they pronounced it equal to the best Ceylon.

Farther back over the hill a number of outcrops, excavations and pits can be noticed, which in varying degree, exhibit the occurrence of graphite, mostly of the disseminated variety in crystalline limestone or in a highly quartzose granite; sometimes pockets of feldspar can be noticed, which contain the graphite in fine scaly particles. Fig. 9.

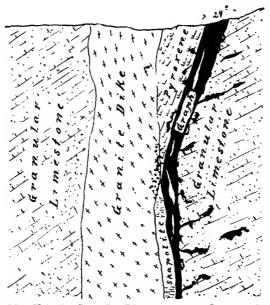


Fig. 9-Graphite Vein in Granular Limestone rear Calumet Station, Can.

This property is very favourably located in the matter of transportation and has other natural advantages; water power can be obtained from Rouge river at a distance of five miles. At the time of this writing a tunnel is being run from the base of the hill towards the main shaft with a view to connecting both by an upraise and in this way tap and explore further, the veins followed in the shaft. A mill has been erected recently on the premises close to the adit to the tunnel. The main building measures 60 x 90 feet. The capacity of the mill is calculated to be about 50 tons of graphite rock per day. A new departure in this mill will be made by the introduction of pebble tube mills for

the cleaning and polishing of the graphite flakes. The motive power in the beginning will be steam, but later electricity will be used, which will be generated from a water power located four miles distant on the Rouge river.

In the township of Amherst, county of Ottawa, several outcrops of graphite of the crystalline variety have been discovered on lots 15, 16, and 17, range 6, containing altogether 300 acres belonging to the Montreal Improvement Co., Limited of Montreal, 90 St. James Street. These properties are situated at a distance, of 12 miles from Jovite station on the Canadian Pacific Railway to the north of Grenville. It is reported that the principal graphite deposit occurs in the centre of lot 16. It consists of a veinlike body of ore of the foliated variety, 25 feet in width, and has been traced for a considerable distance over the properties in a south-east and north-west direction. The country rock is said to be granitoid gneiss. A sample of flake graphite analyzed by Milton Hersey of Montreal gave 73.41% carbon.

### GENERAL CONCLUSIONS ON GRAPHITE AREAS.

In studying closely both the Buckingham and the Grenville districts, we find a great similarity in the character of the graphite deposits; both as to the enclosing country rock and the habitus and main constituents of the vein filling. Commenting on Mr. Osann's observations in this direction it must be mentioned, that the graphite in both localities appears to occur in vein fillings, and is therefore younger than the containing formation, and in a disseminated form through the country rock. The veins generally traverse the latter in all directions independent of its strike, but the majority of them have a north-east direction. Graphite constitutes in most veins the only filling; in others pyroxene, green apatite, scapolite, titanite and wollastonite are associated with the mineral. The country rock has been impregnated from these veins with graphite, and where the same is composed of crystalline limestone, the latter appears to be most susceptible to this influence on account of its loose structure. If the country rock is gneiss this impregnation has been confined essentially to the layers richest in mica, along which also the rock breaks more easily.

On the contacts of the graphite veins the neighbouring rocks have been changed into scapolite and pyroxene, as is characteristic



The New Hundred Ton Graphite Mill of the Calumet Graphite Mining and Milling Co., at Calumet, P.Q.

in the case of apatite veins. At Grenville, the granular limestone has been converted into a mixture of pyroxene, wollastonite and titanite.

From an economic point of view the disseminated graphite is the more important, since it occurs in larger workable deposits, and compared with the vein graphite gives far steadier returns. In the early days of graphite mining, chief attention was paid to graphite occurring in these veins, because it was naturally the purest and most concentrated, and required in the majority of cases little cleaning; but the quantity generally obtained was so small, that it did not warrant the expenditure for extensive development work of these limited and uncertain deposits. Today all the larger mines obtain their principal output from disseminated ore bodies, and the chief attention is directed now to the discovery and development of such deposits.

Summary of Graphite Localities in the Province of Quebec.

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Township of Grenville . . . . . range 2, lot 16, and range 3, lot 16
                                   near Calumet station.
                               range 5, lots 9 and 10.
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Augmentation of Grenville . . range 2, lot 3.

5,

Township of Amherst. . . . . . range 6, lots 15, 16 and 17.

Chatham Gore. . . . . . . . range 4, lot 5.

Township of Buckingham ... range 4, lots 1, 2, 3, 4, 22, 24.

19, 20, 21, 22, 23, 24,

10. 3, 4, 12, 13, 14, 17.

11. 4, 5.

12, 23, 24.

4. lot 47. Township of Cameron .....

Township of Lochaber, adjoining Buckingham on the east.— In this township some of the graphite deposits are of considerable extent, and have been mined at intervals for forty years. They are found on:—

Range 4, lots 28.
" 7, " 10, 24.
" 8, " 23, 24, 25.
" 10, " 22, 23, 26, 28.
" 11, " 21, 23, 24, 25, 26, 27.
" 12. lot 23.

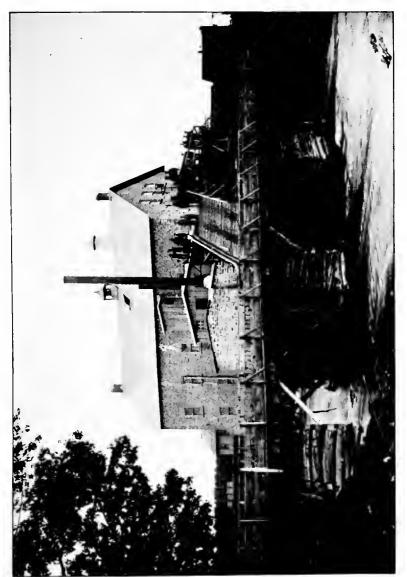
Township of Hull, range 1, lot 9; Wakefield, range 1, lot 7; Wright, range XIV, lot 47; Portland West, range III, lot II. About 30 miles above the High Falls on the Lievre River, on lots 23 and 24, range VI, Cawood and Babiche rapid. Lathburg, range 2, lot II; Wentworth, range III, lots 1 and 2; St. Boniface de Shawinigan, ranges IV, V, VI; on the St. Maurice River, at Point à la Mine, two miles above the Piles; S.E. Provost, range II.

The above list includes all localities, known so far which contain graphite deposits of more or less value. It is likely that with the progress of settlements, especially in the northern parts of that province, other occurrences of graphite will be discovered. It is true, a great many of the above named localities do not warrant expenditure for development work, but it must also be said that there are properties of great value, which await only exploitation by experienced management backed by ample capital.

#### Ontario.

Sir William Logan, in his report of the year 1846, mentions a graphite occurrence on lot 21, range A, township of Westmeath, near the Ottawa river, in crystalline limestone. In his report of progress in 1863, he refers also to "plumbago of good quality in the township of Burgess, occurring in a disseminated form very generally through the Laurentian limestone in the rear of Kingston." He mentions also a plumbago deposit near the outlet of Gold lake, range IX, lot 6, of Loughborough, in crystalline limestone, from 3 to 18 inches in width. The mineral was intermingled with vitreous, translucent quartz, in which portions of pure graphite are sometimes imbedded. Of further localities, lot 18, range IX, of Bedford, and on Bird lake in the same township are mentioned, all of which contain graphite in strata of crystalline limestone.

Many discoveries of graphite have been made since that time



Graphite Mill at North Elmsley, Operated by Water Power.

in the province of Ontario, and some of them have proved of large extent and commercial importance. H. G. Vennor, in his report in 1872-73, describes a deposit at North Elmsley, range VI, lot 21, near Oliver's Ferry, on the Rideau canal. The history of this property is interesting reading. Discovered in the year 1871, the property was worked at intervals by different people; a mill was built for the treatment of the ore, but mine and mill were shut down in The property then lay idle for 18 years until 1893, when Mr. J. Fraser Torrance resumed operations, but only for a short time. In 1901, Dr. R. A. Pyne, of Toronto, took over the property, and in order to test the extent of the graphite deposits in depth, a number of diamond drill borings were made to a depth of from fifty to one hundred feet. These operations were also extended to lots 22, range VI, and lot 23, range VII. In all these borings graphite of the disseminated variety was encountered, sometimes also in pure quality, while some of the cores showed also the presence of apatite. After the termination of this test work, the mine was acquired by Mr. Rinaldo McConnel, of Ottawa, in 1902, who has been conducting mining operations ever since, and has erected a mill for the treatment of the ore.

The mineral occurs here in rocks similar to that of the Grenville series, while the general occurrence closely resembles the deposits of the Buckingham district.

The principal mining operations are carried on at present about 300 yards from the old workings in a south-westerly direction in a bed of crystalline limestone. The principal opening consists of an open pit about 250 feet in length, and a width varying from six to ten feet and a depth from ten to fifteen feet. Most of the openings are made at the crown of an anticline. At a distance of about one hundred feet in a southerly direction from this excavation is another pit eighteen feet deep by thirty feet long, and from four to eight feet wide with a six foot drift from the bottom.

The crystalline limestone contains rusty bands of sillimanite gneiss the dip being generally from 5° to 10° with the exception of the north-east end of the main trench, where the strata dip at an angle of 40°. The ore occurs in a disseminated form as flakes through the limestone and gneiss, sometimes accumulations of the mineral can be noticed suggesting its occurrence in lenticular small masses and pockets. Eruptive rocks are not observed in the fresh excavations, but in the vicinity of the works in a southeasterly direction some graphite can be noticed. There appears

to be no amorphous, columnar or foliated variety; all the mineral seems to occur in a disseminated condition through the rock in form of flakes. A mill for the separation of the graphite from the gangue is located at a distance of three miles from the mine, about one and a half miles from Elmsley station on the Canadian Pacific Railway. The process is dry throughout. The ore after being roasted in a dry kiln to drive off the moisture passes through a Blake crusher and two sets of rolls. Thence it passes through a series of Krom pneumatic jigs, where the separation of the graphite from the gangue is effected. The clean flakes then are polished in buhrstone mills and the product subsequently graded in grading screens. The present capacity of the mill is 10 tons of ore in 10 hours, and it is claimed that an extraction of 10 per cent. of graphite from the ore is effected.

Another property of importance and one which contains large graphite deposits is the Black Donald Mine, in the township of Brougham on the west end of White Fish lake, Renfrew county, belonging to the Ontario Graphite Company. This property comprises lots 16, 17, 18 and 19, range 3. The mine proper is located on lot 17, range 3 and by the present road is 14 miles from Calabogie on the Kingston and Pembroke Railway; the lot was originally acquired by John Moore under a Free Grants Act and on the discovery of graphite Moore took out the Crown patents of the mining rights. Exploration work was commenced here under the direction of the writer in 1895 and actual mining for the mineral with few interruptions has been continued ever since. The graphite deposit is located near the shore of White Fish lake and has a general course of north-east and south-west between walls of crystalline limestone, with a dip of 70° west.

The ore occurs in a vertical vein traversing crystalline limestone and is composed mainly of amorphous graphite; the flake variety is found, however, in small stringers and pockets, as well as in the schistose walls, which are several feet thick. The total carbon content amounts to about 65 per cent. of which about 45 per cent. is amorphous and 20 per cent. flake, the remaining gangue being composed of limestone and occasional seams and pockets of chlorite, locally called "mica". The vein is usually uniform in character from wall to wall, and has a very uniform graphite content. There are locally enriched areas of amorphous graphite, containing as high as 80 per cent. carbon, Low-grade bodies of flake also occur which, taken as a whole, seldom exceed



Interior View of North Elmsley Mill. Part of the Polishing Department.

25 per cent. carbon, and a maximum size of flake of about 8 mesh. The ore shute has been found to increase steadily in width from 16 feet at the eastern outcropping, to 26 feet underground at the extremity of the west drifts, about 400 feet away. The graphite in the walls gradually decreases as the more solid country rock is reached, and when the rock contains less than 15 per cent. carbon it is not mined, as at the present time this percentage is considered to be the minimum quantity for profitable treatment in the refinery.

Owing to the similarity in the specific gravity and the size of the flake graphite and chlorite it is difficult to separate the latter from the graphite in the process of concentration. In consequence, those portions of the deposit carrying chlorite are sorted out, together with the richer portions of the amorphous graphite, to form a product containing not less than 50 per cent. carbon, that is shipped in the lump form for foundry facing purposes. The intermixed gangue is not seriously detrimental to this use.

Pits and cross-cuts have exposed the ore body for a distance of 400 feet. Towards the north it passes under the waters of the lake and to the south it is covered with heavy humus. Borings with the diamond drill gave the following results:—

A borehole close to the edge of the lake from the surface down gave graphite 39 feet, mixed limestone and graphite 6½ feet—disseminated graphite 10 feet—limestone and graphite 7 feet—graphite 1½ feet—feldspar and quartz 2 feet—total depth 66 feet. Another borehole sunk further from the lake gave 15 feet graphite—7 feet, crystalline limestone and 6 feet of graphite.

Intrusions of granite in the limestone in the vicinity of the ore lode are frequently observed and contain sometimes accumulations of a scaly graphite, but of no commercial value. The main shaft is sunk in the north-east extremity of the vein near the lake and has a depth of eighty feet and is ten to twelve feet square. The main level is extended to 200 feet out under the lake and to the south-west for 24 feet. In a north-western direction from the shaft at a distance of 140 feet, the vein was located by an open cut ten feet wide and fifty feet long, and at a point fifty feet further another shaft was sunk to a depth of about forty-five feet. Borings in the bottom of the 80 foot shaft with the Government diamond drill revealed the existence of graphite in a depth of 120 feet from the surface; further progress of the drill

was impeded by a hard flinty rock. The last mining in this portion of underground works has disclosed a width of the vein of 26 feet, the greatest yet met with in the most easterly portion of the stopes. At a distance of 210 feet south-west of the main shaft another vertical shaft of a depth of 34 feet has been sunk. A cross-cut from the bottom runs south 46 feet, and from its face an inclined upraise has been driven west 32 feet to near the surface. There are two other pits sunk at 260 and 300 feet south-west of the main shaft with a depth of 20 and 25 feet respectively. In the latter pit the extent of the ore body was established by diamond drill borings.

In the graphite refinery, which is located near the shore of the lake, the wet process is used. The mill machinery consists of a powerful Blake crusher, a ten stamp battery and two buddles 16 feet diameter by  $2\frac{1}{2}$  feet deep and a number of other appliances. specially constructed for the purpose. The graphite, after having passed through the wet treatment, is then conveyed to a dryer and thence by elevator to bins above the top floor, being afterwards sized in a long series of trommels. The different grades of the flake graphite are then subjected to grinding by buhrstones, which polish the flakes, producing a material valuable for lubricants and foundry mould facings. The power to operate this mill is obtained from an electric plant; the electricity is generated at Mountain chute on the Madawaska river about 21 miles southeast of the mine. The power house installed at this point contains four 20 inch water wheels of 600 h.p. total capacity on the one horizontal shaft direct connected to a 350 kilowatt electric generator. The transmission line is strung with three copper wires of a total length of 36,000 feet for the 3-phase alternating current.

The mining machinery in use includes two boilers, one of 45 h.p. and one, an auxiliary of 25 h.p., a duplex cylinder single-drum hoisting engine and a six drill Rand air compressor. This plant is to be maintained in its entirety as a reserve in case of temporary stoppage of the electric plant.

In the autumn of 1902 a break occurred in one of the drifts of that underground portion situated immediately under the lake which flooded all the workings in connection with the main sha't.

During the spring of 1904, arrangements were made with Mr. Rinaldo McConnell whereby he took over the operation of this mine. A dam was built by him around the break, and the mine

# PLATE XIII.



Black Donald Graphite Mill.



The Black Donald Graphite Mining and Milling Plant Renfrew County, Ont.

was thereby redeemed. Operations have been progressing satisfactorily ever since.

The above deposits are practically the only ones in the province of Ontario, which have been developed and worked to any extent. Many discoveries, however, have been made in different parts of the country, and the most important are mentioned in the following list:—

Township of Marmora, range VIII, lot 13\*—Amorphous graphite has been found partly in small veins, partly in a disseminated form, but no work on the property is recorded.

Township of Blythfield, range IV, lot 13 and 14 near the banks of the Madawaska river.—Graphite is reported to occur in a grayish, rusty gneiss associated with granite and greenish gray pyroxene. The two varieties, amorphous and flaky have been noticed.

Township of Faraday, range I, lot 13.—Several outcrops in limestone and gneiss reported.

Township of Denbigh, range VIII, lot 34, Addington county.

—Graphite of the amorphous variety occurs in layers and patches in a calcareo silicious gangue; an analysis of some of the ore showed 51.67 carbon. This property was worked to some extent in 1903 by Mr. I. G. Allan, of Hamilton, Ont.; a shaft to a depth of 45 feet was sunk and some 150 tons of the ore mined. The ore is silicious in the upper part of the deposit, but is said to improve in depth. An analysis of this ore is found on page 100.

Township of Ashby, range VIII, lot 1.—This property adjoins the Denbigh property on the west. It is reported that some good ore was found on this area, but the shipping facilities are lacking, as the distance from the nearest railway station is between 30 and 40 miles.

Graphite discoveries have also been reported on the following properties:—

Township of North Burgess, range I, lot 10.

" Bedford, range 6, lot 2, range IX, lot 18.

" Uungannon, range XIII, lot 28.

" Loughborough, range IX, lot 6.

 $<sup>\</sup>pmb{\ast}$  From the Reports of the Geological Survey of Canada.

A peculiar occurrence of graphite has been noticed in the district of Stony lake near the village of Apsley.\* A short distance east of Apsley a quartziferous pegmatite dike was seen cutting through crystalline limestone. This dike attracted attention from the fact that it contained considerable graphite as a secondary constituent. The mineral was found to fill numerous cracks in the dike matter and was present in sufficient quantity to have caused some one to search for a workable deposit. graphite is evidently a deposit from liquid matter similar to deposits of anthracite, in which the mineral is found coating crystals of other substances.

## New Brunswick.†

The existence of graphite in this Province has been known since 1839, when Gesner in his report on the Geology of New Brunswick described a deposit near St. Stephen, as a "stratum of graphite or black lead, situated between perpendicular strata of schistose rock. This stratum has been opened and was supposed to be coal." However, no work of importance has ever been done on this property.

In the report of the Geological Survey for 1876-77 Dr. Matthew drew attention to certain graphitic slates in the southern part of St. Stephen, the north part of St. Patrick and near Dumbarton station on the Canadian Pacific Railway. It is, however, only at the latter locality, that they appear sufficiently rich in graphite to give any promise of being valuable. In the black slates, which outcrop on the hill south of this station, there are pockets of graphite sufficiently pure to be available for lubricating purposes or for stove polish.

On the coast of the Bay of Fundy, Lepreau Harbour, a deposit of graphite anthracite was mined some 25 years ago for coal. This deposit consists of a band of black shale and coal, in places twenty feet in thickness, in a formation designated as Devonian. The principal work done consists of several shafts and drifts, the deepest shaft having reached a depth of 140 feet. The thickness of the useful anthracite coal band in the principal working shaft was about four feet, its dip ranging from 80° to 90°. The coal obtained from this deposit burned readily under a good draft, but left 36.88 per cent, of a reddish ash.

<sup>\*</sup> Report of the Bureau of Mines, Toronto, 1899, page 213. † Abstract from the Reports of the Geological Survey of Canada.

Strata containing more or less disseminated graphite occur in connection with rocks of varied age and character in the Province, but are especially distinctive of the upper portion of the Laurentian system as found in St. John County. They consist largely of limestones and in places carry sufficient bodies of graphite to admit of being worked.

The most important deposits, or those which warrant the expenditure for exploitation, are situated near the City of St. John, near to the Suspension Bridge. They were mentioned by Gesner in his report on the Geology of New Brunswick in 1840, in which he refers to the limestone of the hill "containing several veins of graphite, one of which is on the north side of the main street, and is upward of four feet in thickness."

Mining appears to have been commenced on this deposit about 1853, when some 90,000 lbs. of graphite are reported to have been exported. However, the mine was not in continuous operation. After a suspension of work for a considerable time the mine was re-opened about the year 1868, and we learn from the report of the Geological Survey, that in 1871 and 1872 some 6,000 lbs., valued at \$12,000, were exported. After some slight interruption of operations, work was again resumed by Mr. S. S. Mayer, of Carleton, from a point on the land of Messrs. Hazen and Botsford, some 600 yards from the river. After the extraction of a few tons Mr. Best and others resumed mining by sinking a new shaft, in what was considered a position more favourable for working. This was 200 feet north-east from Mayer's workings and four feet from the face of the limestone cliff. At fifteen feet from the surface the deposit, which was concealed at the surface, was reached, and it was found to occur at the contact of crystalline limestone with a trap dike. When first struck the deposit had a thickness of a few inches, but it gradually increased in width with depth until at 50 feet it had a width of from eight to ten feet. A drift from the shaft to the north-east resulted in showing a continuous mass of the material between layers of limestone and trap rock, which here come together and present an unbroken face as far as work was continued.

The graphite is associated in places with pyrite which breaks away readily and leaves the graphite comparatively pure.

All the deposits worked so far near the St. John river possess a close similarity. The deposits of the "Split Rock" mine are found in connection with the crystalline limestones at several points, and it appears that the portions most suitable for mining belong to certain bands of argillitic slate rock, through which the graphite is disseminated. The thickness of these bands varies from one to four feet. Graphite from this mine has a loose slaty structure, with a grayish black color, and a submetallic lustre and black streak. Pyrite is also found in connection with the mineral, and an analysis of the ore by Mr. Hoffman gave graphitic carbon 48.775%—rock matter, 50.058%—hygroscopic water 1.167%.

For several years the ore was shipped in a rough state, but a mill was erected in later years for the cleaning and refining of the mineral.

As a result of the explorations and actual mining work it must be said that the mineral on the St. John river occurs in quantities sufficient to warrant permanent operations, the only drawback at present appears to be the lack of suitable methods to produce a clear, stable article, such as the market demands. Other occurrences of graphite are reported from various localities of the province. Thus in King's county an earthy graphite occurs in beds of some twenty feet in thickness, consisting of dark graphitic shale or slate, with a north-easterly strike, traceable on the course of the outcrop for nearly a mile. Mining operations were carried on for sometime, but it appears, that the percentage of graphite in the rock is too low to warrant its economic extraction.

Other localities where graphite has been found are: Dorchester, Mackerel Cove, one mile east of Goose river on the shore of the Bay of Fundy. Most of these occurrences are in slaty bands and appear to have no commercial value.

#### Nova Scotia.

Graphite is widely distributed over Nova Scotia; most of the important localities are in the island of Cape Breton. But the economic exploitation has so far been of insignificant value owing to the limited and erratic character of the deposits. The rocks in which the mineral is found are of pre-Cambrian age, and consist of crystalline limestones with slates and shales with granitic intrusions. At Glenvale, River Inhabitants, Inverness county, graphite occurs as scaly particles through a coarse red syenite. At Dallas Brook, beds of graphitic shale are associated with limestones and slates and at one time were mistaken for coal strata. Graphitic shale also occurs in the vicinity of Guthra lake

near the French Vale road, and can be traced for some distance in a width of from one to two feet. Some of this shale was analysed in the laboratory of the Geological Survey and gave graphite 38.387%.\* As to the quality of the purified graphite the latter appears to be of fair quality for the manufacture of lead pencils. Its value as an economic product will, therefore, it appears, be dependent very largely upon the cost of its extraction and purification.

Some analyses of the Grendale shales show a great divergence in the contents of graphite. Thus one sample collected by Mr. Fletcher in 1878 gave but 13.965 per cent. carbon; another one from the same deposit gave 31.8%; a third sample taken from the shales of Christmas Island, which is identical with the Glendale occurrence, gave 50.23%.†

On the Salmon river in the county of Guysborough, graphite has been found in black slate near the contact of the gold bearing slates. Of other localities may be mentioned: Parrsborough, Salmon river, Musquodoboit, Hammond's Plain, Fifteen-mile stream, Boularderie island, Gregwa brook and Gill's brook. In these deposits graphite occurs principally in the form of graphite slates or shales, but no mining of importance has been attempted on them. Further occurrences have been reported from West Bay, Grand Narrows, East Bay, and Hunters Island.

### British Columbia.

A very interesting occurrence of this mineral, which, however, was mistaken by the finder for molybdenite, was discovered by Mr. Downie in 1860, at Alkow Harbour, Dean canal,\*\* on the coast of British Columbia. Several fragments of the material were received from him. The largest of these measuring ten inches in length by six inches in width, has a maximum thickness of about three inches, and weighs six pounds twelve ounces. It consists of minute lustrous, dark steel-gray coloured scales and scaly layers of graphite, together with small quantities of pyrite, disseminated th ough a matrix consisting almost wholly of heulandite. An analysis of what was regarded as a fair average sample of the material showed it to contain 23.17% of graphite.

<sup>\*</sup> Report Geol. Survey of Can., 1879-80.

<sup>†</sup> Dr. Ells, Bulletin on Graphite, 1904, page 6. ‡ Gilpin, Report on the Mines of Nova Scotia, 1880. \*\* Rep. Geol. Survey, 1896, page 16 R.

#### Hudson Strait.

On the occasion of the visit of the Diana\* to the whaling station called Black Lead, in Cumberland sound, north side of Hudson strait, in 1897, specimens of graphite from the neighbourhood were obtained. The occurrence of graphite in the various localities above mentioned around Cumberland sound is interesting in connection with the abundance of the mineral at many places among the crystalline rocks on the north side of Hudson strait, and it is a fact tending to show that the rocks around the Sound are also referable to the Grenville series.

#### UNITED STATES.

Graphite deposits are very widely distributed all over the United States, in fact the mineral has been found in nearly every state of the Union, either in commercially useful quantities or as an occurrence of mineralogical interest only. They have been developed in Maine, New Hampshire, Massachusetts, Rhode Island, New York, Pennsylvania, Virginia, North Carolina, Georgia, Alabama, Ohio, Michigan, Wisconsin, Arkansas, South Dakota, Montana, Wyoming, Colorado, California, Nevada, and New Mexico.

Besides these deposits, graphite has been found in small quantities in nearly all the Western States. In many of the states there is considerable variation in the quantity of graphite produced each year, and there is also considerable change from year to year in the number of producing states. Some of them have been constant producers for many years, and these contain the largest and most valuable deposits of graphite that are known in the United States. The following are the producing states in the order of their importance: New York, Pennsylvania, Wisconsin, Michigan, Georgia, Alabama, Ohio, Nevada, and North Carolina.

# State of New York.

The chief deposits from which the principal supply has been drawn are those situated in Essex and Warren counties, the principal mines being those at Hague, Graphite and Ticonderoga. Prof. Kemp† who has made a special study of the graphite deposits in the Adirondacks, divides them into four groups:

<sup>\*</sup> Rep. Geol. Survey, 1898, page 20 M. † Mineral Industry, 1903, page 185.

1,-in pegmatite veins; 2,-veinlets of graphite; 3,-graphite quartzites, and 4.-in crystalline limestone associated with gneissoid strata. In the pegmatite veins the graphite is coarsely crystalline, but is seldom found in such accumulations as to be of any commercial value; associated with it are feldspar, pyroxene, hornblende and quartz. With the exception of the old workings of the Dixon Company, on Chilson Hill, Ticonderoga, the pockets and uncertain deposition of the mineral has rendered mining very uncertain. The graphite occurs in small veinlets of an inch or so in width, and is usually associated with fine quartz. This class of veins does not warrant expenditure for mining, but the graphite quartzites, which form regular stratified members of the sedimentary series, are the steadiest and most reliable deposits from a mining point of view. The quartzites are feldspathic and the graphite, which is seldom pure, but is associated with other minerals like pyrite, occurs in scaly lamellar particles between the quartzites. The percentage of graphite in the rock is from 5 to 15, the mineral being not as coarse as in the pegmatite veins, but it occurs in larger aggrega-Deposits of the above described character are mined near Hague, on the west-end side of lake George, see Fig. 10, by the Joseph Dixon Crucible Co., and in the towns of Dresden and Whitehall, on both sides of South Bay, lake Champlain.

Walcott\* describes the deposit at the mines four miles from Hague as a bed of from 3 to 13 inches in thickness, composed of alternating layers of graphitic shale or schist, which can be traced for over a mile. The garnetiferous sandstones form a strong ledge above and below the graphite bed. The graphite deposit has the appearance of a fossil coal bed, the alteration having changed the coal to graphite and the sandstones to indurated, garnetiferous, almost quartzitic sandstone. The graphite bed is here about 9 feet thick, and is formed of alternating layers of highly graphitic sandy shale and schist.

The graphitic quartzites are treated in two mills, one using a wet and the other a dry process. In the mill at Graphite, the graphitic quartzites are first stamped in California drop stamps, and are then washed in buddles to a state of approximate purity. The final method of purification or concentration is a secret process. In the Lakeside mill the rock is crushed without water and carefully dried to eliminate all moisture, and the concentration is then performed by Hooper's air jigs. Another mill has recently been

<sup>\*</sup> Bulletin of the Geological Society of America X, 1898, page 227.

erected at South Bay, an arm of lake Champlain, about six miles north-west of Whitehall.

The graphitic limestones occurring in the district are coarsely crystalline aggregates of calcite or dolomite with disseminated crystals of pyroxene, graphite and other minerals. These limestones so far have not been a commercial source of graphite, great

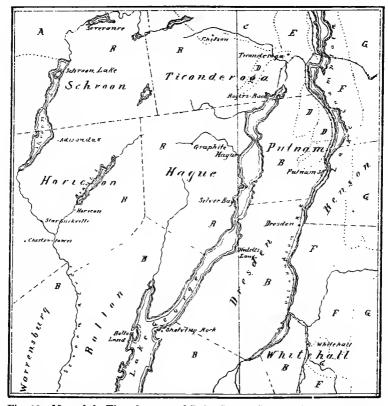


Fig. 10—Map of the Ticonderoga and Lake George Graphite Mining Districts, State of New York. A.—Trachyte. B.—Adirondac Gneiss. C.—Grenville Limestone. D.—Potadam. B. Beckmanstown.

difficulty being experienced in separating the graphite from the mica, with which it is associated.

Kemp describes the graphite deposits near Ticonderoga, N.Y., as true fissure veins cutting the laminæ of the gnessic walls at nearly right angles. The wall rock is a garnetiferous gness, with an east and west strike, and the vein runs at the big mine north 12°

west with a dip of 55° west. The vein filling is evidently orthoclase (or microcline), with quartz and biotite and pockets of calcite. The mineral is also associated with tourmaline, apatite, pyrite and sphene.

It is likely that all the valuable deposits are in the graphitic quartzite, as the lenticular shaped ore bodies in the limestone have proved uncertain. A very large low grade deposit of graphitic quartzite was discovered by Prof. Kemp on the east shore of lake George,\* about three miles back of Hulett's Landing. hanging wall is a very large eruptive, and the vein as at the Hangue mine, seems to have been a line of weakness. The flake of this deposit is very small, and of too low grade to be commercially useful.

Aside from the operations of the Joseph Dixon Crucible Co., there was little graphite produced in New York during 1904.† The mine and works of the Adirondack Mining and Milling Co., on South Bay, near Dresden, were idle for most of the year, as were those owned by the Ticonderoga Graphite Co. The mines are situated near Rock Pond, between Ticonderoga and Schroon, where there is a mill equipped with stamps and buddles. character the deposit resembles the graphite quartzite of Hague, but it is of larger size and somewhat lower in grade.

A new undertaking is the Champlain Graphite Co., which was organized late in the year to develop a deposit of graphitic schist near Whitehall. A mill is now under course of construction. The Silver Leaf Graphite Co. of the same place did not engage in productive operations during the year.

Some attempts to mine graphite have been made on the opposite side of the Adirondacks in St. Lawrence county. and disseminated deposits occur in association with crystalline Some development work was done last year on a prospect near Pope Mills, town of Macomb. The graphite occurs as fine scales in schist, and the deposit is said to be extensive. About 500 tons of rock have been taken out and a mill has recently been completed.

The production of crystalline graphite from New York mines in 1904 was 3,132,927 pounds, valued at \$119,509. There was little change in the output compared with previous years. As the New York graphite is of the crystalline variety, that state appears

<sup>\*</sup> Mineral Industry for 1902, page 347. † Geol. Rep. New York State Museum, 1905, page 29.

perhaps as promising a district for prospecting and developing graphite properties as any in the Union.

#### Rhode Island.

The Rhode Island Graphite Company has been working for several years on a deposit of amorphous graphite near Providence. The graphite occurs mostly in pockets and lenticular masses, and also in fissure veins in granite. Graphitic slate has also been found in the vicinity of fissure veins and contains sometimes from 10 to 20 per cent. of graphite. It is also of interest to note, that in connection with the development of the graphite deposit, a vein of semi-anthracite coal has been opened up. It is claimed that this graphitic anthracite, the purer graphitic portions of which have been mined and used for foundry facings early in the eighties, has peculiar advantages for melting iron and copper ores, and has been used by the Carbon Iron Co. instead of coke with better and more economic results.\*

#### Maine.

There were two graphite mines worked during the summer of 1904, one near Madridt, Franklin county, by the Maine Graphite Co., and another one at Yarmouth, Cumberland county. two occurrences are of interest from both the industrial and the scientific standpoint. The Madrid graphite deposit is located in the town of Phillips, at a short distance from the little village of Madrid. The nearest railroad point is Madrid station, on the Phillips and Rangely railroad. The schist, which together with the intrusive granite, constitutes the country rock in this region, outcrops here along the northern slope of a hill immediately south of the village. Beds of varying composition can be distinguished in the schist and indicate a north-east and south-west strike. The general dip is nearly vertical, although the schist is much contorted. The graphite occurs locally in the schist, always close to the contact with pegmatite, with the exception of the sporadic occurrence of a few slickensided lenses of graphite rock about 18 inches in their longer diameters. At no place seen in the workings, which consisted of an open cut into the hill parallel with the strike of the schist, did the graphite portion of the schist extend more than a

<sup>\*</sup> Mineral Resources of the United States, 1888, page 361.
† Economic Geology, United States Geol. Survey, 1905, page 480.

few inches from the contact. At the point from the face of the cut, where the two bodies of pegmatite are nearest together, the schist is crumpled and crushed into blocks which appear to be largely graphite. However, all of the schist is not strongly graphite at the pegmatite contact. Where the bedding planes can be traced with certainty, it is seen that one stratum may be graphitic and the beds on either side relatively barren, even at the point of contact with the pegmatite. This pegmatite schist contact is well defined, and while the muscovite selvage contains a small content of graphite, the muscovite does not apparently extend into the schist.

A thin section of the best grade of graphite rock collected at the Madrid cut shows this schist to contain small grains of graphite evenly distributed throughout the rock. This is in most intimate association with muscovite, the small folia of the latter being thoroughly interwoven with the graphite flakes and grains. In other bands fine grains of quartz form the matrix of the graphite particles. The schist is beautifully foliated and a glance at either the specimen or the thin sections, leads the observer to overestimate the amount of graphite present. This is due to the fineness of grain and thorough dissemination of the graphite through the rock. Although it is evenly distributed through the rock, the foliated character of the graphite causes the surface of this graphite schist to seem very rich in graphite. The graphite particles are in reality minute, and range from 0.20 to 0.01 mm. in diameter, the average size being less than 0.04 mm.

A sample was collected of the best of the graphite schist as exposed in October in the face of the cut at Madrid. The amount of graphite in this sample was determined in the Survey laboratory, by E. A. Sullivan, as 8.5 per cent.

As to the Yarmouth occurrence, the country rock in the locality where the mineral is found, that is about half a mile from Yarmouth village, is a fissile schist, fine grained, but apparently not at all carbonaceous. The schist is cut by large intrusions of granite, and in one of the large masses of granite graphite bearing pegmatite occurs. This pegmatite has been prospected at several points within 200 yards, and occurs in the form of a dike with an average width of one foot. The pegmatite is for the most part of medium grain, and quartz and feldspar are the principal constituents. Small amounts of mica occur, but only sporadically, while graphite is an important constituent. A few nests of graphite about an

inch in diameter occur in the pegmatite, but most of the graphite is in the form of disseminated flakes evenly distributed through the rock. No definite difference between the content of the graphite near the walls of the dike and that at the centre could be noted.

A chemical determination of the graphite in a representative sample of this graphitic pegmatite was made and shows  $\dot{9}$  per cent. to be present.

# Pennsylvania.

The Pennsylvania Graphite Company in 1897 opened up the Riley mine, near Mertz town, Berks county. The mineral occurs in a fine grained conglomerate or a coarse grained sandstone, which in the vicinity of the mine is weathered to a considerable depth. The deposit is lens-shaped, stands nearly vertical and has a maximum width of 39 feet, thinning out in both directions. The average yield of graphite from the mine is 28 per cent. of the material handled.

Prof. T. C. Hopkins\* contributes the following notes as to the occurrence and production of graphite in the vicinity of Chester Springs, Chester county: "The Philadelphia Graphite Company owns a mine about one mile east of Chester Springs, which was opened in 1897, and was productive in 1898. The graphite is of the crystalline variety, occurring in richly impregnated beds of mica schist, of which the graphite forms about 50 per cent. There are two beds, an upper one about 4 feet thick and a lower about 6 feet thick, which are worked through adit levels. The mineral is trammed to a mill nearby, where it is crushed and washed in a log washer, the washed graphite being further ground and screened into different grades. The product in 1898 realized from \$75 to \$150 per ton.

"At Byers station, a few miles south of Chester Springs, graphite was mined in 1898 by Pettinos Bros., who do not grind or grade the mineral like the Philadelphia Graphite Company, but simply wash the crude material which is shipped to Bethlehem for foundry facings. At Pikeland, about a mile north of Chester Springs, graphite was formerly mined in considerable quantities, and a good crushing and washing plant was erected, but it has been idle for several years. The success of the above mentioned concerns led to considerable prospecting for graphite in the Pickering valley, and some new discoveries were reported, although none

<sup>\*</sup> Mineral Industry, 1898, page 383.

became productive in 1898. The works of Pettinos Bros., at Byers, were destroyed by fire early in January, 1899."

### South Dakota.

In the vicinity of Custer, several promising veins of graphite have been reported. One of these deposits was opened up and yielded, it is said, a high grade product, which is adapted for foundry facings. In width these veins average about four feet. On Castle creek, in Pennington county, about twenty-five miles north-west of Custer, there is a large vein containing about 40 per cent. graphite. On one side of this vein there is 10 feet of graphitic slate. Eight miles north-east of Custer, there are several veins of graphite containing a high grade mineral of the crystal-line variety, which can be easily separated from the gangue.

In a number of states, as in the North Carolina deposits in Wake and McDowell counties, the graphite occurs in schistose rocks. constituting from a small quantity up to 25 per cent. or more of the rock. The occurrence of mica and some silica in these schistose rocks makes it difficult to separate a pure graphite from them. The Georgian graphite deposits, which have been producing rather extensively for the last year or two, are in the nature of graphite shale or slate, containing approximately 13 per cent. of graphite. These deposits are located near Emerson, Barlow county, and the product mined is used as a colorer in the fertilizer trade. The material is not cleaned in any way, being simply pulverized, so that 60 per cent. of it will pass through a 24-mesh screen, and all through an 8-mesh screen. The value of this material is, of course, very low, and in the total production of graphite in the United States, it has increased the tonnage materially without adding very largely to the value.

# Pacific States.

Graphite has been observed in many places throughout the Pacific States and Territories, but it appears that only in California, where its occurrence seems most frequent, have any attempts been made to mine and market or otherwise utilize it in a large measure, the first having been made at Sonora as early as 1863, when 1,000 tons of mineral were extracted, most of it being shipped to England, France and Germany, where it brought about \$100 per ton, a price that afforded the shippers some profit. But the

impossibility of securing here any large quantity sufficiently pure for commercial purposes put an end to the enterprise, the labor of concentrating the crude material, which was largely mixed with slate and other foreign matter, having been expensive. Besides the Sonora deposits, graphite has been found in California at the following places: Near Summit city, Alpine county; on the border of Tomales bay, in the coast range of Marine county; near Fort Tejon, Kern county; at Tejunga, Los Angeles county; at Boser Hill, Fresno county, and several places in Sierra, Plumas, Marin and Sonoma counties.

Graphite has been observed to occur in many states covering the Rocky mountains; thus in the Sierra mountains, Humboldt county, Nevada; in Beaver county, Utah; Albany county, Wyoming; in Gunnison county, Colorado, where it occurs in beds two feet thick, but very impure; in the coal measures of New Mexico, and in Levis county, Washington, about 40 miles from Chehalis.

In the Tumet mining district of Chaffee county, Colorado, a graphite deposit is being developed by the Ethel Gold Mining Company, of Detroit, Mich. Samples that have been examined show this deposit to be amorphous graphite of very good quality. This company is now erecting a mill at its mine for concentration and refining of graphite.

#### Alabama.

Flake graphite is being mined in two localities at Stockdale, Clay county and Taylorsville, Alexander county, and it is reported that some of the deposits are quite extensive.

### Virginia.

Virginia has not heretofore been a producer of graphite, but recent developments in Albemarle and Orange counties, at the base of the Blue Ridge, has opened up promising deposits, which are to be worked by the Naylor-Bruce Graphite Company of Charlottesville, Va.

In the Naylor and the Bruce mine, the graphite occurs among gneisses and syenites, in veins dipping at 45° to the east. They range from 13 in. to 8 ft. wide, and are clearly defined from the foot and hanging walls by clay selvages. The graphite is dense and massive, permitting the extraction of single blocks weighing several hundred pounds. The crude ore, analyzed by Froehling & Robertson, of Richmond, showed 76.28 per cent. graphitic carbon.

The operating company owns 624 acres of land, on which graphite appears and is planning at once to build a factory for refining the product.

#### New Mexico.

Some work has been done on some graphite deposits located eight miles south-west of Raton, Colfax county, under the control of the Standard Graphite Company of New York, and an output of some 65 tons of high grade amorphous graphite has been shipped to Moosic, Lackawanna county, Pa., for manufacture into paint and foundry material. The district about Raton is underlain with bituminous coal, the seams of which vary in thickness from three to seven feet. In the portion where the graphite is found, a layer of lava in the form of a laccolith has intruded itself above the coal seam, and by its heat has metamorphosed the coal to graphite. which assumed a columnar form. The graphite is of the amorphous variety and contains a portion of the silica, which was originally associated with the coal. The vein of graphite approximates a horizontal position and varies in width from three inches to two feet. In many places the graphite is free from rock, but contains an admixture of silica. The graphite vein is cut through by canyons and can be traced for a considerable distance. In one canyon three tunnels varying in length from ten to sixty feet, have been driven on the vein, each showing the occurrence of the graphite to be continuous.

Of other localities, where graphite has been found in economic quantities, may be mentioned: Bloomingdale, New Jersey; Clintonville, New York; Lehigh and Berks counties, Pennsylvania; Salt Sulphur Springs, West Virginia; St. Johns, Toole county, Utah.

#### BRAZIL.

Graphite is known to occur near Miguel de Arassuah, in the state of Minas Geraos, but no attempt has been made to exploit the deposits. Discoveries of graphite have also been reported from time to time in other sections of the country.

OCCURRENCES OF GRAPHITE IN EUROPE, ASIA AND AUSTRALIA.

Graphite deposits are less numerous in Europe, Asia and Australia than on the North American continent, but it appears

that they are more concentrated into a few typical localities, especially in Europe, where their economic occurrence warrants mining operations on a large scale. The most extensive graphite deposits and those which have contributed for over a century to the world's supply of the mineral in Europe, are the deposits in the Bohemian forest, and near Passau, Bavaria. Outside of Europe, there are the occurrences on the island of Ceylon, and in northern Siberia, both of which are famous for their production of a purer article than all the other countries. The geological occurrence of some of the more important deposits of graphite of the world, together with data of first exploitation, are summarized in the following Table III, which is admittedly very incomplete for lack of reliable data, but is of some importance in showing the general tendency of graphite deposits to occur in the Laurentian or pre-Cambrian formation, especially in the gneiss or crystalline limestone series.

TABLE III.

Locality.	Character of Mineral.	Country	Geological Age	Date of First Ex- ploration.
Passau District (Bavaria) Leitzenberg Germannsdorf Pfaffenreut Haasdorf Haar	Amorphous	Gneiss	Laurentian	Year  1450 1550 1730 1780 1791
Great Britain Borrowdale (Cumberland)	•	Porphyry	Cambrian Carboniferous	1540–1560 1840
Bohemia Schwarzbach	Crystalline	Gneiss Mica Schist	Laurentian .	1790
Ceylon	u.	Granite and	Archæen	1806 1847
United States Ticonderoga Rhode Island, R.I. Sturbridge, Mass Sonora, California	Amorphous Amorphous &			1841 1841 1863

Locality.	Character of Mirnəal.	Country Rock	Geological Vge	Date of First Ex- ploration.
Canada			1	
St. Stephen, N.B	Amorphous	Schistose		
- '	_	Rock	"	1839
Charlotte county	"		44	1839
Grenville, Que	Crystalline &		į	1000
, , , , , , , , , , , , , , , , , , , ,	Amorphous	Crystalline		
	ZIMOI PHOUS		Laurentian .	1844
Westmeath, Ont	Crystalling &	Limestone.	Laurentian .	1044
Westineaur, Out		"	"	1045
St. John, N.B	Amorphous		D- O	1845
Buckingham, Ont	u	C	Pre-Cambrian	1851
Duckingham, Ont		Gneiss and		
		Crystalline		
D 461 O 4	a	Limestone.	Laurentian .	1861
Bedford, Ont	Crystalline	46		1862
Loughborough		",	"	1862
Burgess, Ont		"	"	1862
Bay of Fundy, N.B				
	thracite	Amorphous	Devonian	1875
North Elmseley, Ont.	Crystalline	Crystalline	i	
	-	Rocks of the		
		Grenville		
		Series	Laurentian .	1871
Renfrew, Ont	Crystalline &			- 3
,	Amorphous	Crystalline		
	1	Limestone.	44	1890

#### Bohemia and Bavaria.

The graphite deposits of Bavaria are found in a north-easterly direction from Passau, on the Danube, near the Austrian frontier. the gneisses of the Bayerischer Wald being rich in occurrences of graphite. In payable quantities the mineral is restricted to a comparatively small area. Approaching the frontier, granite takes the place of the gneiss and simultaneously with this change the graphite disappears, reappearing further in a north-easterly direction in the gneisses around Schwarzbach, in the Bohemian forest. Thence, following in the same north-easterly direction, the occurrences are traced in varying numbers and importance as far as The Passau graphite occurs in large scales, easily separable from the matrix, and its fine condition furnishes excellent material for the manufacture of crucibles of the best quality. On the other hand, the Bohemian graphite occurs in very minute scales, or in an amorphous earthy form, and is in part applied to the manufacture of pencils.

The most important graphite mines in Bohemia are those owned by the Count of Schwarzenberg, discovered in the year

1790. The deposits were primitively worked in the beginning of the nineteenth century, owing to the very limited demand for the mineral at that time.

In the year 1814 the production was only 20 tons; in 1850 it was 200 tons. From this time on a steady increase is noted until the year 1898, when the production reached its highest record, 11,650 tons. The refineries and other buildings cover altogether an area of about 150,000 square feet, and the number of persons

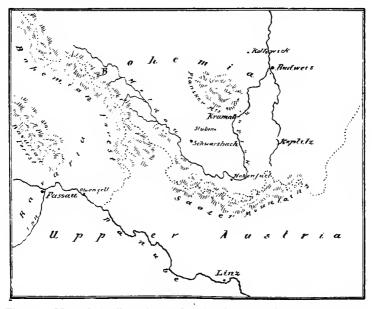


Fig. 11.—Map of the Bavarian and Bohemian Graphite Mining District.

employed is about 700. The works produce not less than 60 different grades of graphite to meet the demands of the modern market. Generally they may be classed into two principal groups, (1) the natural graphite (Naturwaare), and (2) the refined article (Raffinade).

The occurrence of graphite in the Schwarzenberg mines is in the form of longitudinal ore bodies, with impurities of iron pyrite imbedded in a rusty decomposed and partly soft gneiss formation; this vein-like deposit has a general dip of 75°, and extends for over a length of 3,000 feet, and is opened up by sixteen shafts of a depth of from 150 to 300 feet, in connection with about three miles of underground works consisting of drifts and tunnels. Cross cuts

are generally run from the shafts at intervals of 36 feet to the foot-wall of the vein; from these cross cuts, drifts are run along the latter and the deposit opened up by another series of cross cuts, running from the footwall to the hanging wall at intervals of 60 feet. The ore is then taken away between the cross cuts and drifts along the footwall and the spaces filled up with dead material obtained in the course of mining operations.

The run of mine consists of two qualities: one is earthy, soft: the other is flaky, hard graphite. It is interesting to note that the two varieties in these mines seldom occur together, but each of them forms large portions of the vein, so that certain shafts deliver only hard and others only earthy material. The miners are trained in the separation of the earthy graphite by hand in the mine, two qualities being thus produced, one containing over and the other less than 60 per cent. carbon. Most of the work is contract work, the miners getting paid for the quantity of graphite delivered to the shaft. The highest grade of graphite, that is the 60 per cent. and over, is transported to cobbing sheds (Kutterhaus) where little boys separate the ore from the dead material. natural product contains on an average between 70 and 80 per cent. carbon, and is used principally in the manufacture of pencils in Nuremberg and Budweis. The lower grades of the earthy graphite, as well as the flaky varieties, are sent to the refineries.

- O. Bilharz divides the graphite-bearing rocks of Bohemia into two different localities:\*
- 1. Those on the Bohemian frontier to the south, at the foot of the Saazer Mountains.
- 2. Those in the southern part of the Bohemian forest, occurring in genetic relationship with the Bavarian (Passau) deposits of the same material.

In the Saazer Mountains, the clay slates (Phyllite), carry disseminated graphite, sometimes in large quantities, and represent when rich in pyrite a graphitic schist. Of special interest in this locality is the genetic connection of graphite with limestone beds on the older clay slates. The presence of five or six of these limestone beds has been noticed, containing sometimes lenticular aggregations of a comparatively pure graphite. On the surface the graphite appears to have undergone a physical change by the circulation of water; towards depth it becomes much harder and more compact, until it gradually takes the form of graphite schist.

<sup>\*</sup> Zeitschrift fur Praktische Geologie, 1904, page 324.

Deposits of this character have been found near Wachteldorf, also near Swoojanow, where the graphitic schists have a thickness of 12 meters.

More important from an economic point of view, both as to local distribution and to regularity of occurrence, are the graphite deposits in the southern Bohemian forests. The graphite bearing formation commences near Schwarzbach, then extends in a northerly direction over "Stuben," and "Krumau," until it terminates near "Netolic," on the border of the granulit formation of the Plansker mountains. Everywhere in this range the graphite occurs in gneiss, the habitus of which changes in a more or less degree.

A. Pallausch\* describes the graphite deposits between Eggetschlag and Prisnitz, in the Krumau district, as being regularly imbedded along the strike of the gneiss, the latter constituting often a hornblende gneiss. In the vicinity of the deposits limestone occurs and the gneiss, especially in the hanging wall, is mostly decomposed and sometimes changed into a clayey silicious substance, impregnated and discolored by oxide of iron. This soft clay forms the "iron capping" (Eisenhut); if the latter is thick and much discolored the graphite underneath is found generally in a pure state; if there is no iron capping, the graphite is impure and mixed as a rule with kaolin.

The valley of the Olschbach is covered with a layer of one to two meters of peat, covering a bed of one meter of clay, below this comes a much weathered and decomposed gneiss, composed of micagneiss and some limestone and forming the graphite wall of the graphite bed. Fig. 12. At other places limestone beds with alternate layers of gneiss, attaining a thickness sometimes of sixty meters, form the adjacent rock of the deposit. Most of the graphite mined is impure, containing breccias and little pockets of country rock, but pure masses sometimes of large dimensions also occur. The graphite is of the amorphous and crystalline variety, compact and schistose.

Continuing in a northern direction beyond the granulite formation of the Plansker Mountains, the gneiss, which encloses the graphite deposits, is decomposed in the immediate vicinity of the latter, but the habitus of these occurrences is more in the form of seams, while the deposits of Krumau and Schwarzbach resemble longitudinal beds and lenses. Mining is at present confined to the vicinity of the village of Kollowick, situated at a distance of about ten miles in a north-westerly direction from Budweis. In this

<sup>\*</sup> Berg & Huttenmannisches Jahrbuch 37, 1895.

locality the occurrence of graphite in seams predominates, their width varying between 40 and 200 centimeters, both hanging and footwall are composed of soft, much weathered and decomposed gneiss. The extent of the graphite bearing formation is established by boreholes for about  $1\frac{1}{3}$  miles along the strike of the seams, and for a width of about half a mile. The upper portions of the

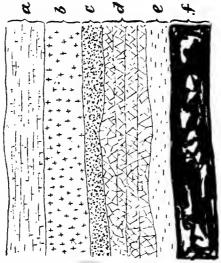


Fig. 12—Occurrence of Graphite in the Valley oi the Olschbach near Kruman, Bohemia. (a) Peat, 6 feet. (b) Clay, 3 feet. (c) Graphitic decomposed Gneiss, 2 to 4 feet. (d) Partly decomposed Gneiss with Hornblende, 6 feet. (e) Decomposed Feldspathic rock. (f) Masses of Graphite in association with Gneiss.

seams generally contain earthy graphite of impure quality, while at depth the finer and purer qualities are met with. The run of mine has a gray black appearance, is composed of fine particles, and for refining purposes is partly subjected to a pneumatic, partly to a washing process.

The occurrence of graphite near Passau is similar to those at Schwarzbach, and is confined to the younger gneiss formation, which contains also another mineral of great importance for the industry of that country, that is kaolin. The extensive distribution of these two minerals is the backbone of a great mining and manufacturing industry, both minerals being employed in the manufacture of crucibles and of chinaware in Obernzell and Hafnerzell, near Passau, employing upwards of 1,000 people. The graphite industry of Passau is several centuries old, and, it is reported, that interesting documents from the beginning of the 15th

century relating to the mining of graphite, the manufacture of crucibles for the "alchymists," and grants and concessions of the "feudal chevaliers and princes," are in the hands of the "Vereinigte Schmelztiegel fabriken and Graphitwerke," in Obernzell, near Passau. The most important mines in the Passau district are at Leitzenberg, Pfaffenreut, Germannsdorf, Haasdorf, Haar, and Hierzing. Most of the proprietors of these mines are peasants. The mineral is found in a depth of from 48 to 130 feet; it does not form a continuous bed, but occurs in alternate layers, nests and pockets in different widths from a few inches to several feet, often suddenly interrupted and cut off by country rock. These beds are seldom horizontal; they dip as a rule between 30 and 40 degrees in a north or north-easterly direction. The boundary planes of the deposits very often constitute a layer of a peculiar compact graphite, showing sometimes slickensided surfaces. The mineral is often associated with mica, in most cases, however, it replaces the latter in the gneiss; the kaolin forms the decomposition product of a peculiar fine grained granitic rock, the feldspar of which is very often found as a secondary product in association with graphite. The gneiss is generally much decomposed and weathered to considerable depth, making mining on account of its soft character very easy, and the graphite occurring in the same way may be justly termed a weathered decomposed gneiss, rich or poor in graphite. In commerce and in the manufacture of crucibles, only such kinds are used, which contain sufficient graphite, generally over 60 per cent., the products exhibiting generally a black brown colour with a deep dark lustre.

Generally speaking, graphite occurs in the Passau district in two different forms:

- 1. As scaly graphite, consisting of smaller or larger scales, resembling mica, comprising sometimes schistose and sometimes compact masses of a loose structure.
- 2. As compact amorphous graphite in earthy masses, having when freshly broken a dull grey appearance, but when rubbed with the fingers a metallic lustre.

The scaly graphite is seldom pure, but contains usually admixtures of feldspar and pyrite. Iron, especially, is a constant companion of the Passau graphite, and it is very often present in the form of limonite, sometimes in such quantities as to render the mineral unfit for use. Very often iron pyrite is found intimately associated with graphite, and in this case the latter is unfit for use in the manufacture of crucibles, as the sulphur does not pass off at

the lower temperatures used. If e.g. silver is to be smelted in these graphite crucibles, the latter have to be subjected first to a red hot heat, in order to eliminate the sulphur, as otherwise the latter would make the metal brittle.

Generally many German works use a mixture of Ceylon and Passau flakes in the manufacture of crucibles, and the product is said to be of excellent quality.

The existence of the Laurentian system in Bavaria and Bohemia, as already stated, has been established by Guembel, both by stratigraphical and palæontological evidence. He finds in Bavaria an ancient gneissic series, estimated at not less than 90,000 feet in thickness, and by him divided into a lower portion, chiefly of red or variated gneiss, which he calls the Bojian gneiss, and an upper portion, distinguished as the Hercynian gneiss. this succeeds a series consisting chiefly of micaceous schists, with hornblendic and chloritic bands, overlaid by what he calls the Hercynian clay-slate formation, which immediately underlies the primordial zone of the lower Silurian system. The prevailing colour of the Hercynian gneiss is grayish. It is very quartzose, often containing black magnesian mica, and frequently having an admixture of oligoclase. Large portions of this gneiss are also marked by the presence of iolite or dichroite, giving rise to a distinct variety of rock, the so-called iolite-gneiss or dichroite-gneiss. Beds of hornblende slate, diorite, and hornblendic gneiss, are also abundant in this series, particularly in the vicinity of the limestone bands, and are often accompanied by beds of metallic sulphurets, and by lenticular masses and beds of graphite, the latter sometimes occurs to such an extent that it can be mined with profit. It is in these strata that the well-known plumbago deposits in the vicinity of Passau are found under conditions closely similar to those of Canada, in the same geological system. The crystalline limestone band near Passau, which occurs in hornblendic gneiss, is from fifty to seventy feet in thickness, and is directly overlaid by a bed of several feet of hornblende slate, between which and the limestone, a bed of three or four feet of serpentine is interposed, and in other parts a layer of nearly compact scapolite, mingled with hornblende and chlorite. The stratified granular limestone beneath contains, among other minerals, serpentine, chondrodite, hornblende, mica, scapolite, garnet and graphite.

#### Moravia.

The most important mines in Moravia are located near Hafnerluden and Pomic. The deposits are here enclosed in a decomposed hornblende gneiss, accompanied by crystalline limestone, and have a thickness of from one to two feet. Besides this locality, there are a number of smaller mines in operation near Altstadt, Schlägelsdorf, Wurben, Müglitz. The graphite in the latter locality is associated with crystalline limestone in gneiss and clay slate, and is said to be of excellent quality. The annual production of all these mines is from 750 to 1,000 tons.

Graphite in northern Moravia occurs in gray to black crystal-line granular Archæan limestone, interbedded with amphibolites and muscovite gneiss, the limestone itself being often serpentinous, in this respect apparently resembling the graphitic portions of the Ophicalcites of Essex county, New York. The material is quite impure, showing on the average about 53 per cent. of carbon and 44 per cent. of ash, the latter being made up chiefly of silica and iron oxide, with a little sulphur, magnesia and alumina. This graphite is regarded as originating through metamorphism of vegetable matter included in the original sediments, the agencies being both igneous intrusions and the heat and pressure incidental to the folding of the beds.

#### Lower Austria.

In lower Austria, graphite is found in the vicinity of Krems. Near Brunn, Taubnitz, two deposits have been under development, the thicknesses of which vary a great deal, two meters being no rarity; but this can suddenly dwindle away to a few centimeters or nothing, or the deposit may be cut up into small patches and pockets or apophyses, which again may coalesce and form a deposit of large dimensions. A great variation is also observed in the qualities; the latter occur from the finest, richest and purest grades to the hardest and poorest varieties, with a content of carbon of from 50 to 83 per cent.

In Styria, graphite occurs near Kaiserberg, partly disseminated, partly in nests or pockets in mica schist, which changes in some places into a true gneiss. Klamberg, in Carinthia, is another locality where graphite is found in a similar formation. The mineral is mostly impure, contains quartz and sometimes small

nests of kaolin. The largest width so far observed does not exceed one meter.

## Northern Germany.

In northern Germany graphite is mined at Friedrichsrode, about seven miles from Gotha; it is shipped mostly to Hamburg, and from here distributed in the form of stove polish. Of other localities where graphite occurs, may be mentioned: Gross-Klenau, near Plossberg; Wildau and Wampenhof, near Arzberg, Hohenburg and Wunsiedel; in the Rhenish-Pfalz, near Didelkopf and Konken, where the mineral occurs in diorite.

In Saxonia, Beck and Luzi found the mineral in the Kreisha section.

# Spain.

Spain produces very fine scaly graphite from its mines near Ronda, in Granada, a few miles from the sea; the article is sent to Holland and to the coast cities, where it is ground and sold as stove polish.

#### France.

In France, graphite is found and mined in the Department des Arriege, Pissie; in the Department Hautes Alpes near Brusian, Vaugansy and Saint Paul, in the Rhône Department.

# Italy.

Italian graphite mines are located in the Pinerolo district of Piedmont,\* and extend from Cumiana on the north along the Cotian Alps to the heights which dominate the Pellice river. The output in 1899 was 9,990 metric tons, valued at \$55,944. The following description of the occurrence and methods of mining is taken from an article by V. Novarese.†

In width the graphite belt varies considerably, being 1,000 to 1,300 feet wide near Giavone, and reaching its maximum breadth in the valley of the Chisone and Germanasca, where it is no less than  $2\frac{1}{2}$  miles wide. At the lower end of the Val Pelliee the breadth is much less and gradually diminishes as one proceeds southward.

<sup>\*</sup> Mineral Industry, 1900, page 380.
† Bolletino del R, Comitato geologico d'Italia, 1898, Vol. XXIX, pages 4-36.

The deposits occur in a garnetiferous mica schist, and range from a few inches to 10 feet in thickness. The mineral has all the properties and characteristic of graphite: black color, semi-metallic, is soft and more or less unctuous according to the greater or less degree of purity, the purest being the least lustrous. On combustion it leaves a silicious ash with mere traces of iron. The schistose material is so intimately mixed with the graphite that it is practically impossible to enrich the poorer grades by mechanical sorting or separation. A number of assays made in the laboratory of the Italian Geological Survey showed from 10 per cent. to 85 per cent. carbon, while the specific gravity ranged from 2.25 to 2.38. The raw product is shipped direct from the mines to the mill, where it is ground and packed. The final product contains about 61 per cent. pure mineral, and has a value of 15s. to 19s. per ton at the mines and about 38s. packed and loaded on cars at Pinerolo.

# England.

The oldest graphite mine is that of Borrowdale, near Keswick, in the county of Cumberland; its discovery was made between the years 1540 and 1560. The geological information regarding this occurrence is very incomplete, due to the fact that the mine has been abandoned for a number of years, and no recent development work has been done. However, from the few reports at present available, it appears that the graphite occurs as minute, fine, scaly particles in compact masses, in a gangue enclosed by a green porphyry.

Mr. J. Postlethwaite\* finds similarities between the containing rocks in Borrowdale and the diamond bearing rocks of Kimberley, South Africa, and considers that the conditions under which graphite was formed in the former locality approached more closely those which gave rise to the diamonds of the latter, than those which originated the graphite deposits of North America.

The principal gangue filling is calspar and quartz, in which pockets and nests of the mineral occur. These deposits sometimes assumed apparently large dimensions, and we hear that "genuine pencils" were cut out from these masses and sold to the European trade. The mine is situated on the slope of a mountain 2,000 feet high; a tunnel at an elevation of 1,000 feet tapped the deposit, and it is said that considerable underground work opened up the vari-

<sup>\*</sup> Quarterly Journal of the Geological Society, Vol. XLVI, page 124.

ous deposits, but no further definite information regarding the latter is available. It appears that this graphite mine in connection with the manufacture of genuine natural pencils for a considerable time was of great importance to England. The government at one time prohibited the export of graphite other than in the form of pencils. Although the mine was only worked for six weeks during the year, and no graphite in the crude state could be exported, yet the deposits, owing to operations being extended for over a period of several hundred years, finally became exhausted, and only the more impure article remained, which could not be used for the manufacture of natural pencils.

### Russia and Siberia.

Deposits of graphite are known to occur in several parts of Russia, European and Asiatic, but the mineral has as yet been exploited only in the Kirghiz Steppes, in Eastern Siberia and Finland. The deposits of Eastern Siberia are especially worthy of The discoverer of these mines is the French merchant J. P. Alibert; he exploited the country tributary to the rivers Oka, Belloi, Kitri and Irkut, for gold and found fragments of pure graphite in the vicinity of Irkutsk. He recognised at once the great economic importance of this discovery and set to work in search of the actual deposits of the mineral. His labors and hardships incident to an undertaking of that nature in a country devoid of civilization and accessibility were great, but finally it is said they were crowned with success. Alibert explored the mountain range of Sojan, and at the summit of the Batougal mountains, at an elevation of 7,000 feet above sea level, and at a distance of 650 miles in a westerly direction from Irkutsk, close to the border of China, he ound a large deposit of pure graphite.\* After mining a large quantity of waste and fragmentary graphite, a very large deposit, of a quality surpassing anything that had ever been found, was The mountain where the mineral occurs has been uncovered. named after the discoverer. Alibert Mountain.

Our knowledge regarding the occurrence of graphite on Alibert Mountain is very meagre, owing to the great inaccessibility; it appears, however, that vein-like deposits of pure graphite cut a granitic, dioritic formation, and that pockets or nests of the mineral

<sup>\*</sup> According to Weinschenk the discovery was originally made by Tunskinsker Cossacks, whose Chief, Tscherepanow, sold the mine for 300 roubles to Alibert in the year 1847.

are also found in the adjacent metamorphic limestone. The graphite in the gangue is columnar and foliated, and has the structure of wood, the fibres generally being arranged at right angles to the enclosing walls. This foliated wood-like structure is of a very pronounced character in the main deposit, and suggested its origin from wooden fibres, as erroneously supposed by Breithaupt. According to a contract made between Alibert and A. W. Faber. of Nuremberg, in 1856, the total output of the mines was sold to this latter firm for a long period, prices as high as 1,700 marks (415 doll.) being paid for 50 kilos (112 lbs.); the best qualities, used in the manufacture of a superior quality of pencil, contained from 97 to 98 per cent. carbon. Recently the mines have been worked only to satisfy the wants of the Irkutsk gold smelting house for the manufacture of crucibles.

According to A. Keppen,\* large deposits of graphite of excellent quality were discovered in 1860 by a merchant named Sidorow, in the north of the Government of Yenissei, along the river Nishni-Toungouska and Koureika. The high grade of this mineral was certified at the Perm Gun factory, in St. Petersburg and in London. The location of these deposits in a distant desert and unpopulated district is the cause of their not being worked.

### Ceylon.

This island is the most important producer of graphite in the world. According to Sir Le Neve Foster, Part IV of the "Mines Report," the world's output of graphite for 1901 was nearly 77,100 tons, valued at nearly £785,000. Ceylon furnishes 29 per cent. of this quantity and 80 per cent. of the value.

Graphite mining in Ceylon has its history. According to A. M. Ferguson, † graphite is mentioned in Singalese letters of the fourteenth century, and in Dutch Government reports of 1675. British records for 1831 give figures of export, which must have commenced between 1820 and 1830, but it was not of any importance until 1834, when it amounted to 129 tons valued at 12,054 rupees.‡ In 1869 the output reached 11.306 tons valued at 889,620 rupees; in 1899, 31,761 tons, valued at 105,366 rupees, and in 1902 it was 25,189 tons valued at 10,516,366 rupees, or £701,098.

<sup>\*</sup> A. Keppen, "The Industries of Russia," Mining and Metallurgy, Vol. IV, p. 92.

† Royal Asiatic Society, 1900, Vol. IX, part 2.

† One rupee=1s. 4d.

General features.—The island of Ceylon is divided into nine provinces, is pear-shaped in form, with a length of 271 miles from north to south, and a maximum breadth of 139 miles. central cone of mountains rising up to 8.296 feet in height, a plain on the north, occupying nearly half the area of the island, and flat or low undulating country on the east, west and south. According to a paper read before the London Geological Society in the year 1900, by Mr. A. K. Coomara Swami, the Island of Ceylon is surrounded by raised beaches, and has been elevated in recent geological times. The gems, for which Ceylon is famous, are obtained from gravels in the Ratuapura district. With the exception of these recent deposits, the island probably consists entirely of ancient crystalline rocks. Graphite occurs chiefly in branching veins in igneous rocks, which are granulites and pyroxene-granulites. The relations to the matrix are held to favor the idea of the deposition of the mineral as a sublimation product or from the decomposition of liquid hydrocarbons.

The mineral character of the rocks of Ceylon present many striking resemblances to the Laurentian strata of Canada, and may perhaps be found to belong to the same system. The Island was, as long ago as 1818, described by Dr. John Davy,\* as made up of feldspathic gneiss and gneissitoid limestone, together with granular crystalline limestone and dolomite, both in mountain masses and in veins, the latter sometimes white and lamellar, enclosing spinel and apatite prisms of yellow mica, cinnamon stone, garnet, yellow tourmaline and zircon, the latter two minerals associated with feldspar and quartz. The lamellar graphite, so abundant on the Island, was regarded by Dr. Davy as the characteristic associate of the gems, spinel, zircon, garnet, etc.

According to George A. Stonier, † graphite occurs in the west and south-western portion of the Island, chiefly in the western and southern provinces, and in Sabaragamuwa. The mineral area is 95 miles long in a north and south direction, with a width of 35 miles at the north and 43 miles at the southern end. well-defined north and south belt 18 miles from the coast which is 5 miles wide at the northern end and touching the coast line at the southern extremity, where it is twenty miles wide. A second fairly well-defined belt is 40 miles in length and 4 miles in maximum width: the payable mineral occurs in veins traversing a normal

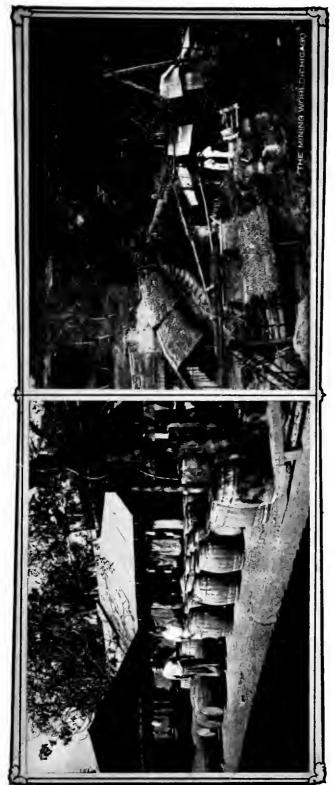
<sup>\*</sup> Institution of Mining Engineers, Vol. 1903. † Trans. Geol. Soc., 1st Series, V, 311.

granulite\* with red garnets. The hanging wall is frequently well defined, roughly polished, and occasionally striated more or less horizontally. The strike varies. In the southern province it is generally meridianal in direction, and in the northern end of the field it is frequently east and west. No evidence of a main vein or a series of lodes has been discovered; the extension of the ore bodies is horizontally limited. Apparently well-defined veins suddenly pinch out, and although one of these has been proved to a depth of 720 feet, it is very doubtful if it is a true fissure vein. The veins vary from a thin sheet to a width of 8 feet. mass of graphite vet discovered is said to have weighed nearly six tons. A vein four inches in thickness is considered to be worth working.

M. Dierschet refers to the country of Kurunegala as the most important locality for the occurrence of graphite. The mineral occurs in the form of veins intersecting granite or closely associated rocks. The country rock is often highly decomposed and then consists mainly of kaolin and similar decomposition products. Three modifications of graphite may be distinguished. The scaly variety, which predominates, the fibrous, which occurs near the boundary plains of the gangue, and the earthy variety. the occurrence of which, however, is rare and confined to the inner portions of the gangue. The mineral has a black-blue, lead gray color, a soft unctuous aspect, and the scaly varieties have a deep metallic lustre. The specific gravity of the pure fibrous varieties is 2.215 and of the scaly 2.235. Moistened with nitric acid and heated on platinum foil over a Bunsen flame, the mineral develops large worm-like forms (see page 12) and is therefore, according to Luzi. a graphite of the first modification of graphite carbon; Weinschenk, t however, recognizes in this behaviour a sequence of higher capillarity, as is generally observed in minerals of a coarse scalv structure.

The graphite of Ragedera occurs in dark easily distinguishable gangues in a light colored garnetiferous country rock, the latter consisting of fresh flinty granulite and pyroxene granulite. The black substance is banded in a typical gangue-like form; it has a columnar habitus near the boundary planes with vertical direction to the latter; further into the inner parts of the gangue, it has a

<sup>\*</sup> Quarterly Journal of the Geol. Soc., 1905, page 590.
† Jahrbuch der Geol. Reichsanstalt, Vienna 48, 231.
† Der Grafit, seine wichigsten Vorkommnisse and seine technische Verwendung, 1898.



A Graphite Mine in the Jungle of Ceylon.

parallel lamellar and scaly structure and finally towards the centre, fine scales and lamallæ are confusedly aggregated together containing fragments of country rock, quartz and other inclusions. The gangues cut the formation transversely and at all angles, their dip being vertical or nearly so; gangues with a flat dip are seldom observed. Often a deposit splits up, forming a great many apophyses, which again may coalesce and form a large deposit from six to ten feet in width.

It seems to be clear that fissures were formed, and then the graphite, quartz, etc., were deposited in the cracks. The quartz may have been derived from a silicious fluid and graphite introduced by sublimation,\* not of the carbon itself, but of hydrocarbons. A deposition of graphite-like material is found in the cracks of the upper layers of coke made in closed ovens; the red hot coke apparently robs the hydrocarbons (distilled from the uncoked coal below) of its carbon, which is deposited as a silvery white layer. A somewhat similar substance is found in the flues of the retorts of gas works. In Bengal, the coal, caked by micaperidotite dykes, sometimes presents a graphitic lustre.

Graphite mining:—† In all, about 300 mines and quarries are at work; and are estimated to give employment to 10,000 persons. With the exception of three mines, they are all worked by natives of Ceylon, and more or less, in a native fashion. European methods have been tried, but have generally failed on account of the inexperience of the manager, or because the company was unfortunate in their site of operations. Graphite mining, like mica and gem winning, is very uncertain, but as a native-owner's costs are low, he can afford to allow a mine to stand idle and await a rise in price. His methods are of two kinds:—If the ground is hard, the chute of mineral is followed as far as water will allow; and, in a few cases, a shaft is sunk to the water-level and the vein worked up to the surface. In soft ground a vertical pit, rectangular in section, is sunk for about 60 feet, and the mineral is followed in a series of winzes 50 or 60 feet deep.

A native usually drives a shaft until he is no longer able to contend with the flow of water in the mine. He then stops working and afterwards drives galleries, and this he continues to do as long as his lamps will burn; but the moment they are extin-

<sup>\*</sup> M. Diersche, Jahrb. d. K. K. Reichsanstalt, Vol. XLVIII, p. 231. † A. Stonier, "Graphite Mining in Ceylon," Institution of Mining and Metallurgy, Vol. 1903.

guished by the gases collected in the gallery, he ceases working in that part and continues upwards, refilling the shafts he has dug, with the debris from the mine. In other cases, instead of sinking a shaft, a large open cutting is made, in which the vein is followed, and galleries afterwards run as occasion may require. There is no system for ventilating the mines, except by small fans worked by hand and the result is that, after a blast, much time is wasted before the mine is sufficiently cleared of foul gases to allow working to be resumed. The great object of the native proprietor is to keep his expenses as low as possible.

The mineral is wound up the shafts and winzes in barrels attached to each end of a locally-made rope (or occasionally an iron chain), which has two or three turns passed around a wooden jack-roll (dabare), 7 feet long and 1 foot in diameter, with iron handles (32 inches long with cranks 13 inches long) at each end; it is worked by six or seven men. The windlass used is frequently not strong enough, and has no ratchet wheel, so that serious accidents may occur in raising and lowering miners.

As to the timber used the native knows nothing of its strength and is quite unable to work out the strain it will stand. He doubtless knows certain timber will resist damp, and is stronger and tougher than other woods; but as the wood is generally green and full of sap, it cannot resist the ravages of damp, as it would do if properly seasoned. The result is that the shafts and galleries are frequently insufficiently timbered.

Round timber is used for securing the sides of the shafts and winzes. The two end-pieces (mukas) are kept in position by two side-pieces (digangs), notched out to fit against the round "mukas" and the "digangs" are kept apart and strengthened by three dividers (one midway and the others at the ends) similarly hollowed out. Horizontal boards, 8 inches wide and 1½ inches thick, and packing or occasionally sticks with ferns (kekilla) are used to jam the set in position. Stulls are not used to support the sets. The kibbles or tubs run on casing-boards nailed to the dividers. The men climb from set to set by the aid of a rope or thin pole lashed to the dividers.

Instead of rope, ladders are frequently used by the miners, and these are made of the roughest materials and frequently tied with jungle rope or ordinary coir yarn. There is no regulated distance between the rungs, and the ladder is placed perpendicularly to the bottom of the pit, and when it is remembered how

highly lubricated the wood must get from the hands and feet of the natives, who have been working plumbago, the great danger they run every time they mount and descend can be well conceived. The timber used is chiefly "alubo" and "hora".

The labor employed is almost entirely Singalese, from Galle. Only men are employed underground, but occasionally women work at the surface. Men earn 8d. to 1s. per day and women receive 3d. to 6d. Tamils from southern India are employed on the tea estates, but they have not taken to mining as a regular occupation.

The mineral is conveyed in bags by coolies or bullocks to a dressing-shed, where it is roughly picked and packed in barrels for transport by road and rail or canal to Colombo or Galle for dressing. The barrels, 22 inches in diameter at the ends and 3 feet long, are made of "hora" wood (Dipterocarpus zeylanicus) and bound with four hoop-iron bands.

Various minerals are dug out of plumbago mines, with which the natives have no acquaintance, and consequently are sometimes thrown away. Pitch-blende, known as a valuable ore of uranium, has been found inside plumbago; pyrrhotite also is found largely in plumbago mines.

Dressing:—On arrival at Colombo, the barrels are opened by Singalese men on an unroofed brick or asphalt dressing-floor (barbacue), rectangular in shape, averaging 39 feet wide and 80 feet long (at Mr. John Kotawala's sheds) and sloping on two sides for drainage. The big lumps are put to one side, and the remainder is carried in conically shaped baskets, 17 inches in diameter at the base and 2 inches deep, and is thrown on to a series of stationary screens (with holes §, §, † and 3-16 inch in diameter, 10 holes to the inch and No. 60 mesh) 3 feet long and 2 feet wide, set at an angle of about 35 degrees from the horizontal. The screened pieces are taken to sheds, near by, which are open at the sides and roofed with cocoanut leaf (Cadjan, Phœnix zeylanica) and women chop them up with small iron hatchets and remove the coarser impurities, such as quartz. The valuable mineral is then placed on boards or on the barbacue, water is added and the pieces are rubbed by hand. The final polish is done by hand on a screen which is placed flat on the ground.

The poor material is reduced to powder by wooden cylindrical mallets (3 inches in diameter and 5 inches long, with wooden handles 1 foot long) or cylindrical beaters (2 inches in diameter

and 14 inches long with the end reduced in size to form a handle) and is hand-picked on sacking, 27 inches long and 20 inches wide, tacked on two strips of wood for carrying purposes.

At some establishments, further concentration is effected by washing in a pit,  $5\frac{1}{2}$  feet long,  $3\frac{1}{2}$  feet wide and  $2\frac{1}{2}$  feet deep. A circular motion is given to the mineral, in a saucer-shaped basket immersed in the water of the pit and the graphite passes into the latter, while the heavier particles remain behind; the graphite is sun-dried on the barbacue. To separate the very fine material, the powdered mineral is placed in a basket, 13 inches long, which is rectangular in section (12 inches by 4 inches) with the corners rounded at one end, and tapering to a line at the other. When the mineral is thrown into the air, the heavier particles fall back into the basket, but the fine graphite is blown forward and falls to the ground.

The mineral despatched to market is classified according to size as lumps, ordinary chips, dust and flying dust, and according to quality as x, xb, good b, b, bc, and p.

Prices rule as high as Rs\* 1,000 a ton for large lumps, and although the price fell to Rs. 700 towards the close of the year 1900, the handsome profits to be earned caused exceptional activity in this form of mining, which was stimulated further by the grant of licenses to prospect on Crown lands.

The district of Kurunegala has 154 plumbago mines. In the southern province 117 acres of land, supposed to contain plumbago, were leased in 1899 for 10 years for Rs. 333,450, and in the central province 126 declarations of intention to open mines were recorded.

The method of working plumbago pits is as a rule unscientific and rudimentary, but European firms are now in the field, and it is to be hoped will prove as keenly interested in introducing improved methods of working, as they are strenuous in their endeavours to secure more favourable terms for working their holdings.

The customs duty on all graphite mined and sold from 1858 to 1868 was  $2\frac{1}{2}$  per cent. ad valorem; from 1846-1857 there was no customs duty by No. 9 ordinance of 1847, and from 1873 to 1883 inclusive, licenses to dig for plumbago cost 10 rupees each, with a royalty of 10 per cent. of the value of the plumbago dug, in the case of the western province.

<sup>\*</sup> One rupee=34.6 cents.

#### India.

Dr. T. L. Walker\* discovered several localities in the northeastern portion of the Kalahandi state, where graphite is met with in parallel bands or veins and apparently of good quality. Travancore remains the only producing state of India. graphite deposits here are similar to those of Ceylon, whose associates form a continuation of the Laurentian strata of South India. Similar occurrences of graphite are found in Coorg and in the hill tracts of Vizagapatam. The Indian graphite like that of Cevlon is regarded as of igneous origin. Prospecting for graphite has been carried out in the Godavari district, Madras Presidency and in the ruby mining districts in upper Burma.

### New South Wales.

Graphite is being mined by the Walcha Graphite Syndicate 26 miles north-east of Walcha.† The mine lies on the sides of a steep spur overlooking the deep canyon of Blue Mountain creek. The graphite occurs in a Eurite dyke, generally micropegmatitic and crowded with spheroidal segregations of graphitic material. The dike sweeps across the main trend of the spur in such a manner that its contents are naturally proved to a depth of at least 400 The country rock is an acid granite, and the dike is strongly marked over 250 or 300 vards of outcrop. It varies in width from 6 feet at the northern end to 30 feet on the southern side of The graphite in the spheroidal kernels is excessively the spur. The kernels vary from ½ inch to 1 inch in diameter, and they occupy from 40 to 50 per cent. of the dike mass. The kernels contain from 20 to 25 per cent. graphite. The mineral is uniformly distributed throughout the dike, and large quantities are extracted.

There is also an important deposit consisting of a bed of graphite-bearing shale of about 6 feet in thickness, five miles to the eastward of Undercliff Station; in the county of Buller. This bed is inclined at an angle of about 45° and is interstratified with schists, sandstones and clay-stones, which are probably of carboniferous age. It is situated close to the junctions of these sedimentary rocks with intrusive granite. The graphite, it ap-

<sup>\*</sup> Geol. Survey of India, Gen. Rept., 1900-01, page 12.
† Mineral Industry, 1904.
‡ Pittman, Mineral Resources of New South Wales, 1901, page 371.

pears, cannot be used for purposes for which this mineral is generally employed, but tests made with larger samples demonstrate that it can be used with advantage as a foundry facing.

Of other localities may be mentioned: Cowell creek, the head of the Abercrombia river, where it occurs in association with quartz, iron pyrites and pyromorphite; in small radiated masses in the granite at Dundee, in New Valley and near Tenterfield; at Panbula near Eden, in quartz; at the Cordeaux river near Mount Keira, and at Plumbago creek near the junction of Timbara creek, county of Drake.

## Queensland.

The principal deposits are at Mount Bopple\* in the Maryborough district, where the graphite has been found sufficiently pure for commercial purposes; and where mines are now being worked with a view of supplying the demand which has been created for it.

The Mount Bopple graphite mines are situated on the range close to the North Coast railway between Gympie and Maryborough, being about three miles south-east of Netherby, and about the same distance north-east of Gundiah. The highest point on the Bopple range is Beacon Peak, 1,900 feet above sealevel, on which has been erected a trigonometrical staff. The peak known as Mount Bopple, 1,800 feet high, is the most northerly one on the range, and is about three-quarters of a mile distant from the Beacon Peak.

The two peaks, together with Mount Gundiah, which is about a mile south of Beacon Peak, are connected by a low range having a north and south trend, and on both their eastern and western sides numerous steep and rugged spurs lead down to the flat, country at their bases. It is on the spurs leading down from the gap between Mount Bopple and Beacon Peak that the graphite-bearing belt of country is situated.

Burrum coal measures are the prevailing sedimentary rocks around the Bopple range, and their intrusion by igneous masses has resulted in the strata being much crumpled and faulted.

Hornblende andesite is the predominating igneous rock on the eastern side of Mount Bopple, and in several places it is seen in contact with the coal measures. With other varieties of ande-

<sup>\*</sup> B. Dunstan, Report Geological Survey of Queensland, 1906.

site it is also exposed in many localities to the north of the range in the direction of Tiaro, about four miles distant, and about a quarter of a mile west of Tiaro, in the banks of the Mary river, a natural section shows its association with coal measures. The intrusion of these igneous masses appears to have disturbed the sedimentary rocks throughout the district, and to have altered seams of bituminous coal to anthracite or graphite.

The occurrence of aplite was also observed at the graphite mines on the western slope of the range. At Simpson's shaft this rock is found in the graphite in the form of small veins about an inch in thickness, which taper out to leaders, and gradually change to a plumose mica. Occasionally the hornblende of the andesite is found in the form of black cleavable crystals about an inch in diameter, and, no doubt, the rumor that wolfram is found in this locality is due to the presence of this mineral

On the western slope of the range the bituminous coal has been altered entirely to graphite, while on the eastern slopes the coal, by almost imperceptible stages, has been changed to semi-bituminous coal, anthracite, and graphite. In one locality the graphite rests on anthracite, with six inches of graphite clay shale between the beds of graphite; anthracite, shale, and associated rocks being quite uniform and apparently undisturbed.

The anthracite is very lustrous and of good quality, but the bands are on an average only about an inch in thickness, whilst the bed of graphite, six inches above, is dull and earthy, and shows no perceptible division into bands of varying quality. The beds above and below are shales and sandstones, and there are no intrusive rocks in direct contact with either the graphite or anthracite. Probably the anthracite has been a coal of good quality, and that the graphite originally has been carbonaceous matter mixed with carbonate of iron. The intrusive masses of hornblende andesite in the vicinity might have induced the change from bituminous coal to anthracite at the same time that the carbon contained in the ironstone was changed into graphite. Subsequent leaching would have removed most of the iron, leaving a residue containing about 50 per cent. of graphite carbon.

The work of exposing and developing the graphite deposits on the western slope of Mount Bopple are now proceeding at Simpson's mines, and a shaft has been sunk to prove the seams which are exposed on the surface.

At the first landing in the shaft a sample was stripped from

a seam of graphite four feet thick, and which on analysis yielded 27.10 per cent. of carbon and 69.20 per cent. of ash. At the second landing a 3-foot seam of graphite was sampled, and was found to contain 32.28 per cent. of carbon and 62.40 per cent. of ash. Another sample was taken from a 2-foot seam at the third landing which yielded 18.32 per cent. of carbon and 76 per cent. of ash. Other samples were taken from positions lower down in the shaft, and gave results somewhat similar to the above. The whole of the analyses of the samples taken are tabulated below.

The seams which were sampled aggregate eleven feet of graphite, having an average composition of 25.59 per cent. of carbon and 69.30 per cent. of ash. Other bands of shale, aggregating 24 feet in thickness, contain a smaller percentage of graphite, while there is about ten feet of shale in which graphite is not present. Sections which have been exposed in the vicinity of the above workings show that other graphite seams are present, interbedded with graphitic shales and sandstones, but their quality has not been ascertained.

The inclination of the strata in the upper portion of Simpson's shaft is at an angle of about 45°, but nearer the bottom the angle of dtp is much steeper, and in one place is nearly vertical.

	TABLE OF	ANALYSES	OF MOUNT	BOPPLE	GRAPHITE.
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No.	Moisture at	Mixed	Ash	Silica	Composition of Ash		Alkalies, etc. (by dif- ference.
v.H.C.	Carbon	Asn	Silica	Alumina	Lime		
1	5.70	27.10	69.20	61.80	30.20	1.90	6.10
$egin{array}{c} 1 \\ 2 \\ 3 \\ 4 \\ 5 \\ 6 \\ 7 \\ 8 \\ 9 \\ \end{array}$	5.32	32.28	62.40	58.30	35.80	1.70	4.20
3	5.68	18.32	76.00		35.60	1.30	5.00
4	4.46	22.14	73.40	55.60	37.20	2.70	4.50
5	5.26	25.74	69.00	50.00	25.00	2.00	4.00
6	5.22	27.98	66.80	59.80	35.10	1.40	3.70
7	2.50	16.10	80.10				
0	3.20	16.30	78.00				
10	$\begin{array}{c} 3.50 \\ 2.80 \end{array}$	15.60 46.30	79.50 51.10				
11	1.10	74.10	24.00	Ash is m	ainler ailiaad	a of A1	
12	2.40	10.80	82.80	Ash is in	ainly silicat	e or Ar	umma.
13	6.20	72.70	21.00				
14	7.00	44.25	48.75				
15	4.40	90.90	4.60				

# Japan.

Graphite has been produced at various times in five provinces in Japan in the prefecture of Gifu, but the amount is insignificant in all but Hida, where the Kawaimura mine has produced some graphite for a number of years.

# CHAPTER III.

## ORIGIN OF GRAPHITE.

There is hardly any other chapter in geology, in which so many theories have been advanced, so many errors committed as in the study of the origin of graphite. For a very long time graphite was generally regarded as purely organic in character; for since carbon is one of the constituents of animal and vegetable life, many authors regarded graphite like coal as of vegetable origin.

The late Sir Wm. Dawson of Montreal estimated the aggregate thickness of graphite in one band of limestone in the Ottawa district as not less than from 20 to 30 feet, and he believed that there is as much carbon in the Laurentian as in equivalent areas of the carboniferous system. He compared the pure bands of graphite with beds of coal, and he held that no other origin can be imagined than the decomposition of carbon dioxide by living plants.

Sterry Hunt,\* the eminent Canadian geologist, gives his views regarding the formation of graphite as follows:—

"The presence of graphite in veins implies its separation from solution at an elevated temperature, and in this connection the curious researches of Brodie have shown that this form of carbon is possessed of singular chemical properties and affinities, which, when further studied, may serve to explain its solution and crystallization. Meanwhile, the observations of Pauli have established that when hydrate of soda, mixed with cyanid of sodium, is heated with nitrate of soda to incipient redness, the carbon of the cyanid separates from the liquid mass in the form of graphite. Pauli, moreover, suggests that native graphite may have been separated from certain carbon compounds by a process analogous to this. (Philos. Mag. 4, XXI, 541). The direct transformation into graphite of carbonaceous matter cannot, however, be doubted by geologists, and such an hypothesis is therefore untenable for the stratified graphites. This reaction described by Pauli is nevertheless instructive, as showing that graphite may be

<sup>\*</sup> Geology of Canada, 1866, page 222.

separated from solutions at a temperature not higher than that at which, according to Sorby, the minerals which accompany it in the Laurentian veins have crystallized, although we cannot, in the formation of these veins, suppose the intervention of the same chemical reagents as in the experiment of Pauli.

"Graphite may undoubtedly be formed at much higher temperatures; its occurrence in cast iron is well known, and Brodie. who obtained, by dissolving a graphitic iron in acid, four per cent. of lamellar graphite, found it to be identical in physical characters with that met with in nature. Jacquelain also, by the decomposition of sulphuret of carbon in contact with metallic copper, at 800° centigrade, obtained, together with sulphuret of copper, amorphous graphite. Starting from this experiment Jacquelain suggests that native graphite may have originated from the distillation into the fissures of rocks, of volatile hydrocarbons, which have there, by a decomposition similar to that which takes place in contact with the walls of coal-gas retorts. given rise to a deposit of carbon, that has assumed the form of graphite (Cosmos, June 23, 1864). Graphite, when ignited with carbonate of lime, gives rise to carbonic oxide, and, under similar conditions, reduces iron from its oxide to the metallic state. even decomposes the vapor of water at a red heat. We are hence led to regard the graphite of bedded rocks as having been formed by the alteration of coal and similar earbonaceous matters, at temperatures below redness; while its subsequent translation into the veins, and its deposition in a crystalline form, together with various other minerals, has been effected under conditions which, although imperfectly understood, probably included aqueous solution at a temperature not far below a red heat."

The theory of the formation of graphite from carbonaceous matter originally present in organic form may perhaps be applied to graphite of bedded rocks and disseminated through limestone, and it is evident that the same process, whereby the limestone was converted into marble, may have been the cause of the metamorphism of the carbon into graphite. But as far as the vein graphite is concerned, this view cannot be upheld, since we find the mineral in the form of fissure veins in intrusive rocks like granite, diorite, etc. However, it is a well known fact that coal, which is itself of organic origin, has in some cases been converted into graphite through metamorphic agencies. The deposits of Newport afford a good example of such transition and Prof.

Newbury\* mentions an occurrence of this nature in the coal fields of southern Mexico, at Sonora: "All the western portions of this coal field seem to be much broken by trap dikes, which have everywhere metamorphosed the coal and converted it into anthracite. At the locality examined, the metamorphic action has been extreme, converting most of the coal into a brilliant, but somewhat friable anthracite, containing three or four per cent. of volatile matter. At an outcrop of one of the beds, however, the coal was found converted into graphite with a laminated structure, but is unctuous to the touch and marks paper like a lead pencil. The metamorphism is much more complete than at Newport (Rhode Island), furnishing the best example yet known to me of the conversion of a bed of coal into graphite."

Another graphite deposit, which according to Walcott has been derived from a bed of fossil coal, is that occurring four miles from Hague on lake George in the Adirondacks, described on page 55.

That graphite can be formed without the aid of vital force is shown by its presence in cast iron, where it crystallises out on cooling in the form of bright metallic scales. This was observed by Scheele as early as 1778. Cast iron is a compound of metallic iron and a small amount of carbon. In the molten condition. however, it can dissolve a large quantity of carbon, up to as much as 4 per cent. of its weight. This excess crystallises out as graphite, when the metal cools. The coarsely crystalline gray pig iron owes its peculiar properties as well as its appearance to the presence of graphite, and when this form of iron is dissolved in acid, scales of graphite remain as an insoluble residue. Graphite has also been found in meteoric masses, which so far have not exhibited any traces of either plant or animal life, as for instance in the meteorite, which fell in 1861 at Cranbourne near Melbourne, and this meteoric graphite is, according to Berthelot, identical in properties with iron graphite. We may thus conclude that the meteoric mass in which it was found had been exposed to a very high temperature.

Weinschenk† has shown that carbonaceous particles in clay slates have been converted into graphite by the metamorphosing influence of intruded igneous rocks. The observations of Weinschenk indicate that the graphite deposits of Styria in

<sup>\*</sup> School of Mining Quarterly 8, 1887, page 334.

<sup>†</sup> Zeitschrift fur Praktische Geologie, Jan., 1903.

the Alps have been formed through the intrusion of a central boss of granite by metamorphism and he bases his theory on the following points:—

- 1. The crystalline characteristics of the slates show no connection with the degree of compression. Slates, which are but slightly dislocated, appear highly crystalline when they are near the granite; others, which are intensely folded and which show transverse slatiness, appear entirely elastic when they are at a greater distance from the granite.
- 2. The transformation of coal to graphite appears also to be independent of the degree of dislocation; it is, however, confined to the immediate vicinity of the granite. Receding from this granite, the place of the graphite is taken by an anthracite, which breaks up on heating, showing in this a characteristic which is particularly prominent in coals affected by contact metamorphism.
- 3. The granite itself near its confines shows an increase of silica and alkalies, also distinct slatiness and porphyritic structure.
- 4. In the slates themselves, bedded intrusions of granitoid rocks occur. The general habitus of the latter clearly bespeaks their nonsedimentary nature. They are petrographically identical with the apophyses, which are so abundant in the entire chain of the Central Alps, taking the form of extensive beds in the slaty rocks, and that of genuine dikes or veins in those which are less slaty. Their development shows sometimes a fine grain; at other times a coarsely grained structure. They are, however, evidently intrusions in the rocks which carry them and are as a rule distinguished by the presence of a certain amount of tourmaline.
- 5. The complete identity in the petrographic type of the rocks at present under discussion, with the central granites of the Alps, speaks for a practically contemporaneous intrusion of both. As the larger number of granite bosses in the Alps are evidently of much younger geological age, it may not be out of place to assume for the occurrences under discussion at least a post-Carboniferous age.
- 6. The very extensive occurrence of talc in the neighbour-hood of the Styrian graphite deposits, as well as the occurrence of talc in the Alps themselves; furthermore, the presence of large deposits of magnesite in the limestones is utterly inexplicable

without the assumption of volcanic agencies. Such intense Chemico-Geological processes are only conceivable as the sequence of volcanic activity and, furthermore, wherever such occurrences are found to any extent, large masses of granite have invariably accompanied them closely.

As to the occurrence of graphite in volcanic rocks there appears to be no doubt that the graphite has been formed by alteration of carbon fragments, which were enclosed in the molten magma during its eruption. For this reason, we seldom observe an even distribution of the mineral through the volcanic rock and fragmentary occurrences as small nests and pockets are the rule, as for instance, in the diamond bearing rocks at the Cape, in the socalled "Blue-ground." In this connection it may be mentioned that the meteorites exhibit much analogy in their composition with these diamond bearing rocks and very often contain a large percentage of graphite, especially so if the meteor is composed mostly of iron. In some of these iron meteorites small concretionary masses of graphite can be found, and it is very likely, that the mineral has been formed through precipitation and subsequent crystallization out of the molten metal while cooling. may be mentioned, that in a few cases diamond is a companion of graphite in meteorites, and that sometimes larger or smaller crystals of diamond have been entirely changed into graphite in meteoric iron.

As to the occurrence of graphite in the Laurentian formation it must be said that the general habitus and association of the same with granite and diorites seems to speak decidedly against its organic origin, at least as far as the graphite in veins and apophyses is concerned, and in this respect they resemble very much the occurrences near Passau, Bavaria.

G. O. Smith describes two deposits in the State of Maine (see page 58), one in Madrid, Franklin county and one in Yarmouth, Cumberland county, and comes to the conclusion that the source of the carbon crystallized into graphite was presumably in the original sediments from which the beds were formed.

The field observations and microscopic study indicate clearly a difference in the character of the graphite in the two occurrences described. The Madrid graphite is exceedingly fine grained and of the variety that is often called amorphous graphite, although the minute particles are in reality crystalline, possessing all the luster of the larger flakes of the Yarmouth graphite. This lack of simi-

larity in size of particles and the differences in form of occurrence are suggestive if not indicative of wholly different modes of origin.

The presence of graphite in the Madrid locality, at the contact between an intrusive mass of pegmatite and somewhat carbonaceous schist, at once suggests a contact origin for the graphite. The relative concentration of the graphite at the contact indicates that the processes active in the formation of that mineral were connected with the intrusion of the granitic magma rather than with the dynamic forces which have affected to some extent the rocks of the region. The source of the carbon thus crystallized into graphite was presumably in the original sediments from which the beds of schist were formed. This inference is based on the variation in content of graphite in adjoining beds even at the pegmatite contact, a variation believed to express the original difference in percentage of carbon in the successive layers of muddy sediments. The possibility is recognized, however, that certain parts of the schist may have exercised a selective influence in the concentration of the graphite, just as it is noted that these adjoining beds contain varying amounts of tourmaline, which doubtless originated from the pegmatite magma. The graphite at Madrid is believed to be the product of the conversion and concentration of carbonaceous particles of sedimentary origin through the agency of the heated vapors issuing from the intrusive rock magma now consolidated as pegmatite.

In the Yarmouth occurrence there is no evidence of any source of the carbon of the graphite other than in the molten rock itself, which intruded the granite. The graphite is as much an essential and original constituent of the pegmatite dike as is the quartz or the feldspar. The graphite crystallized possibly later than the feldspar, but plainly earlier than the quartz, and like these minerals was of magmatic origin.

According to the theory of Weinschenk,\* the source of carbon forming graphite veins must be sought deep down in the earth. Weinschenk ascribes the formation of graphite in these deposits to the probable action of volcanic gases, probably of carbon monoxide and carbon monoxide compounds of the Cyanogen group, and of iron in the presence of carbon-dioxide and water, all of which having ascended along lines of fracture with the eruptive rocks, were deposited as graphitic carbon through chemical agencies. Gruner showed in 1869 that the action of carbon monoxide at a

<sup>\*</sup> Ibid.

temperature of 300°C. on iron ores gave rise to the formation of graphite, and it is very likely that graphitic carbon has been formed in a similar way in these veins after the manner of many of our economic minerals. In all these chemical processes the co-operation of organic substance is excluded, whether as an original constituent of the rock mass or as of a secondary origin. In the Canadian Laurentian deposits the association of graphite with eruptive rocks can be clearly noticed, and judging from the exploration so far conducted, it appears that the presence of granitic dikes has had a decidedly beneficial influence on the formation of the graphite in the adjacent crystalline rocks, for we find very often in approaching a granitic dike an increase of the mineral in the gneiss. A similar relationship has been noted in the Passau graphite mines; it is there a well known fact that the large scalv graphite of very pure quality and the richest and largest number of graphite lenses or pockets are invariably found at the contact of the granite masses with the gneiss.

The distribution of the graphite through the rock mass in the vicinity of the veins is supposed to have taken place through planes and fractures of the rock in contact with the vein, and in some cases accumulations are encountered which are of even higher value than the vein itself. Osann,\* in his treatise on the Canadian graphite deposits, refers to the alteration of enclosing rocks into scapolite and pyroxene as characteristics in the case of apatite veins. In this way masses have been formed, which are very similar to the pyroxeneites of the apatite region. At Grenville, he noticed the alteration of granular limestone into a mixture of pyroxene, wollastonite and titanite. In both cases minerals have been formed. which are essentially the same as one is accustomed to observe in limestones, which have undergone contact metamorphism. contact metamorphism, according to Osann, can only be explained by the assumption that the limestone has been penetrated by gases and vapors from the neighboring eruptive magma and upon further cooling, perhaps also by solutions, and that in this way the materials foreign to limestone have been introduced. The assumption of a similar process in the formation of graphite veins, Osann says further, is most reasonable. The occurrence of apatite and graphite veins in such close proximity in the province of Quebec, and exhibiting so much in common geologically, shows that they have had a similar origin, and Osann, like Weinschenk, supposes a pro-

<sup>\*</sup> Rep. Geol. Survey of Canada, 1899, page 780.

cess of volcanic action after the cooling or solidifying of the eruptive magma. It is only necessary to remember here the occurrence of graphite in apatite veins, and conversely of apatite in graphite veins. The latter is reported from Ceylon in all geological descriptions.

As to the disseminated form of graphite through the limestones of Canada, there seems to be very little doubt that the same is derived from carbon originally present in the rock, probably of organic origin, and that by the same process of metamorphism, by which the limestone was converted into marble, this graphite has also been formed. However, the graphite occurring in veins has certainly nothing in common genetically with this disseminated graphite, and as above outlined the source of its carbon, according to Weinschenk, must be sought deep down in the earth.

#### CHAPTER IV.

# COMPOSITION OF GRAPHITE ORES.

All graphites, whether natural, artificial or refined for commercial purposes, contain almost invariably volatile substance and foreign rock matter. The latter, in the case of natural graphites, consists chiefly of silica, alkaline earths, oxides of iron, titanium and chromium: less frequent the sulphides of iron, copper, nickel and cobalt. Of all these impurities, silica, clay and oxide of iron are the most commonly met with. A certain English graphite gave a considerable residue of oxide of chromium, contaminated with a little oxide of iron, while some of the Canadian Laurentian graphites show minute quantities of cobalt and nickel, probably due in some cases to the presence of pyrite according to Dr. Sterry Hunt, and to pyrrhotite according to Dr. Hoffman. In several kinds of graphite a small quantity of ammonia compounds was found.

Pure graphite as outlined in a previous chapter is a chemical individuum, a modification of carbon, and in a dried condition contains no water. The presence of chemically combined water, which is often found in natural graphites, appears to be due to an admixture of clayey substances or of other hydrated silicates.

Hydrogen enters very often into the composition of graphite ores, and in the following table the investigations of several well known chemists in that direction are laid down:-

TABLE 4.

Locality.	Carbon	Hydrogen	Ash	Authority
		l		
Ticonderoga	99.87	0.11	0.02	Luzi*
Crystals of Ticonderoga	99.86	0.12	0.02	"
Cevlon	99.82	0.17	0.01	"
Passau (Bavaria)	99.93	0.65		"
Siberia	99.89	0.10	0.01	44
Canada (locality not specified)	86.8	0.50	12.6	Regnault
"	76.35	0.70	23.4	""
"	98.56	1.34	0.2	"
"	99.5		0.68	44
Siberia	89.51	0.60	10.4	"
Artificial graphite	96.97	0.76	0.4	"
Canada (locality not specified)	98.56	1.34	0.20	Cloez‡

<sup>\*</sup> Berl. Ber. 1891, 24, 4085.

<sup>†</sup> Dammer, Handbuch der anorganischen Chemie, Vol. II, Part I, page 260. ‡ Ann. de Chim. et de Phys. (4) VII, 450.

#### CANADIAN GRAPHITE ORES.

As mentioned in a previous chapter, the workable deposits of graphite in Canada contain the amorphous and the crystalline or flaky variety, the latter more frequent than the former. The amount and nature of the impurities generally met with in the graphite ores vary as much as the contents of carbon. In the majority of cases the percentage of carbon in a graphite is all that is asked, but in comparing various ores for specific purposes, it is not less important to know exactly the nature of the accompanying gangue and the percentages of the same.

The purest Canadian natural graphite has invariably been extracted from veins, and this graphite, if properly hand sorted, need not be subjected generally to mechanical refining; carbon as high as 99.815 per cent. has been found in some of the natural vein graphite of Buckingham.

The disseminated or flake variety, however, that is the one which so far has furnished and still furnishes the bulk of the ore for the production of graphite, is comprised for the greatest part of gangue, the graphite itself constituting but a small part of the ore. As a rule the percentage of graphite in disseminated ores, as experience has shown, to be economically useful, should not fall below 5 per cent., but one case is known in New York State, where a mine, owing to an elaborate system of refining, can treat ore containing as low as 3 per cent. flake graphite with a profit. The percentage of graphite generally met with in disseminated ores, from which so far the bulk of the Canadian graphite has been extracted, according to figures before the writer, may be put down from 7 to 30 per cent.

The case is entirely different with amorphous ores; while perhaps 5 per cent. flake ore might under certain conditions pay to work, owing to the comparative ease of extraction of the graphite flakes, it is problematic whether an amorphous ore containing 30 per cent. of graphite would pay to work, on account of the most difficult problem of concentration, and attendant loss of graphite in the tailings. Special emphasis is laid on these two conditions of graphite ore, because the uninitiated as a rule cannot understand why two ores of the same contents of graphite, and even of the same gangue, are so different in the degree of their economic usefulness. As to the composition of the ore obtained from veins, the graphite in many cases entirely fills the latter;

the impurities commonly met with are gneiss, feldspar, calcite, wollastonite, green apatite, scapolite, quartz, titanite, pyroxene and pyrite. Canadian disseminated ores contain in the majority of cases either gneiss or crystalline limestone or a mixture of both; less frequent are quartz, feldspar, pyroxene, wollastonite, pyrite, mica, tremolite, diorite, etc. In the Black Donald mine on White Fish lake, county of Renfrew, amorphous and flake graphite occur together in a deposit not homogenous throughout. Much of the ore contains calcite sometimes in such proportions as to render portions of the deposit useless. A similar combined flaky and amorphous ore is found in the township of Blythfield near the bank of the Madawaska river.

The amorphous graphite ores frequently consist of graphitic shales, slates or anthracite like material as in some parts of Nova Scotia and New Brunswick. An amorphous ore of a highly calcareo-silicious gangue is found in Addington county, township of Denbigh.

In the following tables 5 and 6 some analyses are given of a number of graphite ores taken from various localities of the Dominion.

Sample 1 is taken from a bed of graphite averaging 8 feet in width and crossing lot 28 in range VI and lot 27 in range VII, and may be considered a fair average of the deposit. It occurs in scales and is so closely and evenly distributed through the rock as to almost entirely mask the nature of the latter.

Sample 2 is taken from an outcrop. This deposit apparently consisted of a much decomposed and brownish yellow rock, containing the graphite as scales in an evenly distributed form. There is no calcite present, but hydrochloric acid with the aid of heat dissolved large quantities of alumina, iron, lime and magnesia and small quantities of silica and manganese.

Sample 3 is taken from a large bed of disseminated graphite. The latter is pretty evenly disseminated in scales through the rock and contains some calcite and small quantities of pyrrhotite. Hydrochloric acid with the aid of heat dissolved large quantities of alumina, iron, lime and small quantities of silica, manganese and magnesia.

ABLE 5.

Authority.	Dr. Hoffman, Ottawa.	v	3	"	Milton Hersey, Analyst,	Montreal.  Dr. J. T. Donald, Mont-	real.	F. Cirkel, Montreal. Dr. Hohmann, Dussel-	dorf, Germany.	F. Cirkel, Montreal.
Hygro- scopic Water	0.044	1.740	1.176	0.135	:	1.167		2 34 1.38	1.12 0.84 0.96	1.02
Total Soluble & Insol. Rock Matter.	72.438	75.875	75.026	69.349		50.058		13.54 12.87	19.25 25.83 27.58	71.03 53.68
Rock Matter not Soluble in HCl.	54.899	56.408	53.741	66.874		:		: :	17.84	64.60 42.22
Rock Matter Soluble in HCl.	17.539	19.467	21.285	2.475	:	:			7.99	6.43 11.46
Graph- itic Carbon.	27.518	22.385	23.798	30.516	96 to 99.5	48.775		84.12 85.75	79.63 73.33 71.46	28.15 46.25
Kind of Graphite.	t 28, Dissem. & scaly.	Dissem	Dissem. & scaly.	Disseminated		:	Ren- Lump (dense &	faky)	33 3 3 33 3	Disseminated
Locality.	1—Buckingham, Que., lot 28, Range VI	R. VI. 3—Buckingham Que lot 28	R. VIII Gue, lot 23	R. VI.  Bucking ham. lot 28. R. VI.		5—Falls at the mouth of St. John river, St. John, N.B.	6—Black Donald Mine, Ren- frew county, Ont	" — <u>L</u>	8— " 10— " 11—Ruskingham 145 91 £ 99	R. VII

## MONOGRAPH ON GRAPHITE.

	Authority.	Geol. Survey of Canada, 1879, p. 1 and 2. Dr. Hoffmann. Rept. of Mines Department of Nova Scotia.	Dr. Hoffman. Geol. Survey, Can., 1894, p. 2, R.
	Hygro- scopic Water		1.167 86 33
	Total Soluble & Insol. Rock Matter.	61.613	50.058 1.167 27.86 48.33
	Rock Matter not Soluble in HCI.		
	Rock Matter Soluble in HCl.		
	Graph- itic Carbon.	38.387 13.965 31.8 50.23	48.775 72.13 51.67
	Kind of Graphite.	Graphitic shale.	Amorphous
***************************************	Locality.	Guthra lake, Nova Scotia  Glendale Glendale, N.S. Ghristmas Island, N.S.	M.B  Marmora, Ont., lot 13, R. VIII Amorphous  Addington county, twp. of Denbigh, lot 34, R. VIII Amorphous

TABLE 5—continued.

Sample 4 is taken from a deposit of disseminated graphite, imbedded along the stratification of the country rock, which here consists mostly of quartz and feldspar. Small veins of a twisted fibrous graphite cross the country rock and the deposit and vary in thickness from a few inches to  $1\frac{1}{2}$  and 2 feet. A solution of mineral matter in association with the graphite, effected by hydrochloric acid, contained small quantities of alumina, iron, manganese, lime, magnesia and some traces of silica.

Numbers 6 to 10 were taken from a vein in crystalline limestone, 12 feet wide, containing the mineral in an evenly distributed state, sometimes assuming a dense appearance, and sometimes small flaky accumulations. In samples 7 and 8 the flakes were separated from the dense graphite by passing the latter through a sieve of very fine mesh. Nos. 11 and 12 represent average samples of disseminated graphite through gneiss in the proximity of small narrow apophyses and branches of graphite veins.

The following table 6 gives the composition of the ash from various samples:—

#### Notes.

No. 13, the structure of this graphite was massive, dense, and was made up of broad and thick laminæ. The color was dark steel gray with a metallic lustre. The sample contained here and there thin seams of foreign mineral matter. As explanatory of the presence of nickel and cobalt in the ash of this graphite, it may be mentioned that the pyrites from veins in the Laurentian rocks were long ago found by Hunt to occasionally contain cobalt and nickel and sometimes in notable quantity; in the present instance, however, it is more probable that their presence is due to pyrrhotite, also a nickeliferous and cobaltiferous mineral, and which has been shown to be present in the beds of disseminated graphite occurring in the same locality

No. 14 was taken from the centre of a vein; it had a lenticular shape and contained a core of corresponding form, consisting of orthoclase and calcite. The structure of the graphite was compact and columnar. The graphite breaks readily in the direction of the structure into more or less angular aggregates, each aggregate being made up of thin, narrow foliæ of very uniform width. The foreign mineral matter was evenly distributed through the structure of and as a film upon the graphite, so that on incineration the residual ash formed a tolerably perfect cast of the fragment

									P.	Percentage of Ash.	e of As	ė.					
Locality.	Kind of Graphite	Specific Gravity	Graph- itic Carbon	Volatile Matter	Ąsh	Silica	Alu- mina	Sesqui oxide of Iron	Sesqui- oxide of Man- ganese	Lime	Mag- nesia	Pot- ash	Soda	Prot- oxide of Copper	Oxide of Nickel or Cobalt	Total	Authority.
No. 13-*Buckingham, lot 21 and 22, R.	R. V.																
7.44 .* D. ol-in ol-om	foliated	2.2689	99.675	0.178	0.147	56.08	11.12 13.27	13.27	0.35	8.9	6.74	2.2	2.8	99.0	8.0	100.854	100.854 Dr. Hoffman, Ot-
lot 27, R. VI Vein graphite	Vein graphite columnar	2.2679	97.626	0.594	1.780	45.73	10.82	1.23	0.47	34.74	0.95 0.52		5.40	:	:	99.871	tawa. T
3, R. II Vein graphite	Vein graphite foliated	2.2714	99.815	0.109	0.076 55.08	55.08	8.5	18.31	0.31	7.7	2.02 4.78		2.97	1.16	0.12	100.945	:
1, R. VI Vein graphite	Vein graphite columnar	2.2659	99.757	0.108	0.135		60.80 10.04 16.72	16.72	0.87	4.40	3.88	1.03	1.05	1.94	0.29	101.020	
17—Black Donald Mine (Renfrew) Flaky and a-	Flaky and a-		84 19	4 66	11 99	17.6	14 00	16.49		7 08	10					1	To December March
			84.06		19.75	3.90				10.05 44.03	16.96	:::	: .: : .		: : :	100.05	Crescent Steel Co. F. Cirkel, Montreal
†Buckingham	:	2.2813	78.48	1.82	19.17	65.00	25.1	6.2	o I	)	:	·		:	:	98.00	98.00 Mène, †Paris.
high), lot 34, R. VIII	lot 34, R. Amorphous.	:	76.12	, : ·	:	5.70	:	1.28	:	16.20	0.70	:	:	:	:	:	Thomas Heys & Son, Toronto.
· * Rept. Geol. Survey of Canada,	Survey of Can	ada, 1876	1876-77, page 489-506.	489-506.					† Jahrt	uch fur	Chemie	sche Te	hnol., 1	† Jahrhuch fur Chemische Technol., 1869, 231			

employed. The color of untarnished foliæ was dark steel gray with a metallic lustre.

No. 15, specimen taken from a pure graphite occurring in lenticular bodies in limestone; it weighed about 8 kilograms and was of great purity. The exposed faces of the laminæ had become tarnished with a reddish brown colored film, but apart from this it contained very little foreign matter. The structure was massive, dense, made up of broad and thick laminæ, closely interlocking each other at diverging angles, thus presenting a radiated arrangement, the sides of the vein forming the basal line. Color, dark gray with metallic lustre.

No. 16, sample had a massive structure made up of narrow laminæ, interlocking each other at such an angle as to present an almost columnar appearance. The specimen was very pure and contained no readily perceptible foreign matter.

No. 17, a sample taken from a massive accumulation of graphite in limestone. Impurities were hardly perceptible, but with the aided eye, small particles of calcite could be detected. The specimen had a somewhat schistose laminated structure, some of the lamallæ being of appreciable size and hard texture. Fine minute flakes cover in some places the larger lamellæ, while the earthy material constitutes the greater part of the specimen. The lustre is highly metallic with a brownish tinge.

No. 18, this sample had a massive structure with partly earthy partly crystalline appearance, but the quantity of flakes was small. Calcite was the principal impurity and appeared to be evenly distributed. The sample represented a fair average of a vein of 10 feet in width.

#### Conclusions

Judging from the foregoing analyses, it appears that the vein graphite is the purest and requires, when found in compact form, hardly any dressing. The flaky varieties taken from veins show generally a very satisfactory content of carbon, in most cases over 75 per cent. In the case of the Black Donald graphite the largest quantity of impurities contained therein is represented by carbonate of calcium. A great variation in the contents of carbon will be noticed in the various samples of the disseminated graphite, and it is very difficult, almost impossible, to obtain from one and the same deposit two samples which have the same or nearly the same contents of carbon. It is evident, therefore, that as the

greatest bulk of the mines consists of disseminated graphite, the difficulties in producing at all times commercial grades of a fixed standard are manifold, and the production of the latter is one of the most essential conditions in graphite mining, since even with rich and inexhaustible deposits neglect in that direction proves almost in every case disastrous to the undertaking.

# Graphite From Foreign Countries.

The following table 7 gives the comparative analyses of graphite from different foreign localities. These determinations were made by M. Mene,\* analytical chemist, on specimens sent to the Paris International Exhibition in 1866. All samples received the same treatment, one part of the pulverized specimen being dried at a temperature of 120° C., and the other part was fused in a platinum crucible, the remaining ash was analysed in the usual way.

<sup>\*</sup> Mene, Jahrbuch fur Chemische Technologie, 1869, page 231.

TABLE 7.

	• :	5			٥	Composition	of Ash	in per cent	ند
Localities.	Specific	Volatile Matter	Carbon	Ash	Silica	Alumina	Oxide of Iron	Lime and Magnesia	Alkalies and Loss
Cumberland, First quality.	2.3455	1.10	91.55	7.35	52.5	28.3	12.0	6.0	1.2
Ordinary quality	2.2379	3.10	80.85	16.05	:		:	:	:
Ordinary, in pieces	2.5857	2.62	84.38	13.00	62.00	25.0	10.0	5.6	<b>†</b> .0
Ordinary, in powder.	2.4092	6.10	78.10	15.80	58.5	30.5	7.5	3.5	:
Passau, Bavaria	2.3032	7.30	81.08	11.62	53.7	35.6	8.9	1.7	ଟା ଟା
	2.3108	4.20	73.65	22.15	69.2	21.1	5.5	2.0	1.9
Mugrau, Bohemia.	2.1197	4.10	91.05	4 85	61.8	28.5	8.0	0.7	1.0
, ,	2.2279	2.85	90.85	6.30	:	:	:	:	:
Fagerita, Sweden	2.1092	1.55	87.65	10.80	58.6	31.5	7.0	0.5	၃) ၃)
Ceylon crystals.	2.3501	5.10	79.40	15.50	:	:	:	:	:
Ceylon, commercial quality.	2.2659	5.20	68.30	26.50	50.3	41.5	?! \$	0	:
Gulf of Spencera, South Australia	2.3701	2.15	25.75	72.10	:	:	:	:	:
, , ,	2.2852	3.00	50.80	46.20	63.1	58.51 10.82	4.5		3 9
Altstadt, Moravia.	2.3272	1.17	87.58	11.25	:	:	:	:	:
Zaptan, in Lower Austria	2.2179	2.20	90.63	7.17	55.0	30.0	14.3	:	2 0
Praz, Bohemia	2.3309	2.07	82.68	15.25	:		:	:	
Ceara, Brasilia	2.3865	2.55	77.15	20.30	0.62	11.7	2.8	1.5	0.0
Buckingham, Canada.	2.2863	1.82	28.48	19.17	65.0	25.1	6.3	0.5	1.2
Madagascar.	2.4085	5.18	69.02	24.13	59.6		8.9	?i	9.0
Pissie, Dep. Hautes Alpes.	2.4572	3.20	59.67	37.13	68.7		×	1.5	6.0
	2.3280	2.17	72.68	25.15	:	:	:		
Brussin, Dept. Rhône, France	2.2029	0.28	92.00	7.72	:	:	٠	:	
Vaugnesay, France.	2.1050	0 13	94.30	5.57		:		:	:
Sainte Paul, Rhône Dept.	2.3656	0.17	92.50	7 33	:	:			:
	:	0.14	93.51	6 65	:			:	
Schwarzbach, Bohemia	2.3438	1 05	88.05	19.90	62.0	5X.51	6.3	1.5	1.7
Ural	2.1795	0.72	94.03	5 25	64.2	24.7	10.0	8.0	6.3

Weger (according to Muspratte—"technische Chemie") gives the following analyses of graphite from various localities.

TABLE 8.

Locality.	Ash %	Carbon %	Authority
Borrowdale, Cumberland, England	13.3	86.7	Karsten.
orio wazie, edinocrizita, Englana	46.6	53.4	Prinsep.
Ceylon in crystals	1.2-6.00	94.0-98.8	44
" cleaned	18.5	81.5	"
" crude	37.2	62.8	"
" in crystals	3.9	96.1	Knapp.
" India, from Himalaya	28.4	72.6	Prinsep.
Bustletown, Pennsylvania	4.6	95.4	Vanuxen.
Siberia, Alibert Mountains	3.4	96.6	Wagner.
" "	3.8	96.2	
" "	8.5	91.5	44
First Quality	3.9	96.1	Weger.
Schwarzbach, Bohemia	12.5	87.5	Ragsky.
Hafnerluden, Moravia	57.0	43.0	",
Rana, Lower Austria, crude	41.3	58.7	"
" " cleaned	51.1	48.7	"
" " ground	49.5	50.5	66
" crude for cru-			
cibles	73.7	26.3	. "
Kaiserberg, Steiermark	57.8	42.2	Forstel.
" for crucibles	35.6	64.4	"
Hafnerzell, near Passau, Bavaria	58.0	42.0	Ragsky.
" "	65.1	39.4	Berthier.
"	52.9	47.1	Knapp.
Wunsiedel, Bavaria	0.33	99.77	Fuchs.

Dr. Hoffman\* gives the following composition of graphite from Ceylon and Ticonderoga:—  $\quad \bullet$ 

TABLE 9.

Locality.	Spec. Grav.	Volatile Matter %	Carbon %	Ash %
Ceylon, vien graphite, columnar Ceylon, vein graphite, foliated Ceylon, vein graphite, columnar Ceylon, vein graphite, foliated	2.2671 2.2664 2.2546 2.2484	0.158 0.108 0.900 0.301	99.792 99.679 98.817 99.284	0.050 0.213 0.283 0.415
Ticonderoga, N.Y., U.S.A., vein graphite, foliated	2.2599	1.191 0.818	96.656 97.422	2.153

<sup>\*</sup> Rep. Geol. Survey of Canada, 1876-77, page 507.

The average composition of graphites from different localities is given by Kretschmer\* in the following:—

TABLE	10.
-------	-----

	L	oc	al	it	y.						,	Volatile Matter %	Carbon %	Ash %
Ceylon												5	62.5	32.5
Moravia Steirmark												1.63	53.0 73.3	1 44.0 25.0
Bohemia													43.9 42.67	53.5

These figures are the average values of a great number of analyses of larger quantities of run of mine and commercial graphites.

In carefully selected portions of the gangue the contents of carbon in the Ceylon graphite in the better qualities is as high as 83.5 per cent., and in the best quality produced, 97.5 per cent. In the Bohemian graphite mines apart from the hard graphite masses, which compose the greater bulk of the deposits, a very soft graphite occurs, which is used in its natural state in the manufacture of pencils and crucibles, the contents of carbon being from 80 to 85 per cent.; in Schwarzbach in the Ida mine a very fine scaly graphite is found, the contents of carbon being as high as 90 and 95 per cent. This graphite is exclusively used for the manufacture of crucibles. The quantity of natural graphite in those parts of the deposits, where it occurs, comprises only from 15 to 20 per cent. of the total mass available for mining purposes.

F. R. Ragsky† gives the result of comparative analyses of graphites from Hafnerluden, Moravia, from Schwarzbach in Bohemia, and from Passau, Bavaria:—

TABLE 11. composition of ash in percentages.

Locality.	Ash	Silica	Iron oxide	Alumina	Lime
Schwarzbach, I. qual Passau, commercial Hafnerluden	% 12.5 58 57	% 5.1 26.4 49.2	% 1.2 6.5 0.8	$\begin{array}{c} \% \\ 6.1 \\ 25.1 \\ 7.0 \end{array}$	0.1 

In addition to the above tables the following analyses grouped according to localities will be of interest:—

<sup>\*</sup> Donath, der Graphit, 1904, page 73.

<sup>†</sup> Jahrb. Geol. Reichsanst, 1854, page 201.

Italy: Sesini\* analysed two specimens from Monte Pisano and found:—

TABLE 12.

	Ι	II
Hygroscopic water	% 5.52 48.88 2.65 42.95	% 1.95 18.67 1.83 77.55

England: Graphite from Borrowdale contained 88.37% carbon, 1.23% hydrogen and 9.8% ash. The latter contained 5.1% silica, 1.0% alumina, and 3.6% iron oxide.

<sup>\*</sup> Gazz. chim. ital., 1895, page 25.

TARLE 13

SIBERIA.\*

Locality	Carbon	Siliea	Oxide of Iron	Alumina	Lime and Magnesia	Volatile Matter	Sulphur	Authority.
	%	%	%	%	%	%	%	
Stepanovsky—Mine I	36.06	37.72	4.02	17.8	1.20	3.20	Trace	1
Stepanovsky—Mine II	33.20	43.20	3.05	15.42	4.03	4.03	0.04	} Ncm*
Bagoutal-Mountains	38-40	38-40	4-5	13-14	1-2.8	1.2-3	:	Hepworth Collins*

\* Donath, Ihid, pages 71-75.

Greenland: Nordstrom\* analysed graphite from Karsok, taken from a vein 8 to 10 inches thick in clay, sand and conglomerate, and found it to contain, after drying at  $120^{\circ}$ C., 93.7 to 97.7% carbon, .7% hydrogen and 4.9% ash.

Bohemia: Kretschmer† gives the following table of average compositions of Bohemian graphite:—

TABLE 14.

1.—NATURAL GRAPHITES.

	Carbon	Ash	Volatile
Mugrau mines.	%	%	%
	33.308	65.985	0.707
Schwarzbach mines—  Hard Soft Soft Soft Soft Soft Soft Shadana Shadan Shadana Shadana Shadana Shadana Shadana Shadana Shadana Shadan Shadan Sh	51.629	47.255	1.116
	66.021	32.904	1.075
	87.597	11.315	1.088

#### 2.—REFINED GRAPHITE.

19		Carbon	Ash	Volatile.
Mugrau (Mokra) " " " " " " " "	1	% 96.125 84.388 66.150 60.927 59.212 59.089 48.395	% 2.605 15.192 33.717 38.493 40.612 40.473 50.930	1.270 0.420 0.133 0.580 0.176 0.438 0.775
" " "	8	52.453 50.963 49.058 48.771 47.801	49. 50. 51.	547 037 942 229 199

<sup>\*</sup> Donath, ibid, page 71-75.

<sup>†</sup> Donath, Ibid, page 73.

The following table gives the complete analyses of two Bohemian graphites:—

TABLE 15.

	C	SiO <sub>2</sub>	$\mathrm{Al_2O_3}$	$\mathrm{Fe_2O_3}$	CuO	$\mathrm{FeS}_2$	MnO
Mugrau	57.35	$14.64 \\ 21.07$	$8.38 \\ 8.57$	2.28 2.30 6.19 4.94	0.01 trace	1.89 6.96 0.79 0.65	trace trace

	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	H <sub>2</sub> SO <sub>4</sub>	H <sub>3</sub> PO <sub>4</sub>	H <sub>2</sub> O
Mugrau	0.60	0.74	1.47	0.18	0.08	0 072	2.55
	0.26	0.48	1.49	0.26	trace	0.046	3.55
	0.23	0.53	0.44	0.04	0.72	0.16	3.65
	1.01	1.03	1.545	0.105	1.12	0.08	2.60

Graphite of Hardtmuth used for the Bessemer process is composed, according to von Juptner of the following:—

PERCENTAGE COMPOSITION OF ASH.

Carbon	Ash	Silica	Oxide of Iron and Alumina	Lime	Alkalies
83.77	%	%	%	%	%
	16.23	45.96	52.04	1.91	. 09

Styria: C. von Hauer and C. John\* analysed several samples of graphite from Rottenman. These contained 86.0, 27.4 and 26.6% ash, and 14.0, 72.6 and 73.5% carbon respectively. In other samples of the same locality the constituents of the ash were determined as follows:—

No.	Ash	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	$\mathrm{Al_2O_3}$	CaO	S
1 2	70 32.5 60.9 73.8 53.2 24.9 60.0 31.0	7/ 21.50 27.50 39.50 33.50 17.00 39.50 21.50	7,0 1.75 5.00 4.00 11.50 3.50 7.24 3.50	7.50 25.50 6.75 5.75 3.25 11.75 4.25	% 1.25 2.50 2.25 2.50 trace 1.50 1.25	% 0.31 0.24 9.38 trace 0.22 0.38 trace

TABLE 16.

Five samples of graphite from the mines of von Mayr were analysed by Dr. Margoshes,† and gave the following:—

	I	п	III	IV	v
Carbon	% 70.15	% 23.7	% 64.6	. 72.9	72.5
bound. Sulphur Ash. Composition of the ash—	$\begin{array}{c} 6.32 \\ 0.09 \\ 23.53 \end{array}$	$5.07 \\ 0.52 \\ 71.23$	$5.97 \\ 0.10 \\ 29.43$	$egin{array}{c} 4.32 \ 0.16 \ 22.78 \end{array}$	$4.36 \\ 0.11 \\ 23.14$
Silica	14.10 7.23 0.10 0.27 0.83	35.35 28.60 0.50 1.50 5.28	17.03 10.31 0.40 0.45 1.24	13.97 7.36 0.26 0.35 0.84	14.88 7.03 0.22 0.40 0.61

TABLE 17.

Moravia: Prof. Hoenig of the University of Brunn has analysed a number of Moravian graphites and gives the following percentage compositions:—

<sup>\*</sup> Jahrbuch der Geol. Reichsanstalt 25-159.

<sup>†</sup> Laboratory for Chem. Tech. Geolgy, Academy at Brunn.

# COMPOSITION OF GRAPHITE ORES

Locality	Water	Carbon	$ m SiO_2$ and $ m Al_2O_3$	СаО	MgO	w	$sO_3$	Alkalies
	9.75	26.52	7% 17.81	2.50	20.2	0 991 991	%0	25
Lichotin, near Kunstadt Keenov	5.41 9.73 5.57	7.69 28.14 46.62	22.49 20.86 16.42	Trace Trace	11.0s 0.77	0.06	9999	. 61 . 62 . 64 . 68
earthy scaly ground refined	3.02 5.39 4.76 3.04	36.98 52.33 34.41 38.02	17.96 9.98 22.0	0.57 0.68 0.98	0.93 1.88 Trace 0.45	0.06		4.79

### MONOGRAPH ON GRAPHITE.

New South Wales: The following analyses of samples of graphite have been made at different times in the laboratory of the Department of Mines in Sydney\*:—

TABLE 19.

Lime	96.0
Alum.	15.93
Silica	27.96
Moisture	8.77 7.35 8.66 7.90 7.33 6.61
Ash	58.25 59.58 63.50 63.84 46.27 75.68
Carbon	46.28 34.40 31.76 28.60 33.83 47.12
Description of Mineral	Felspathic Graphite Plumbago clay Inferior Graphite Inferior Graphite Graphitic Clay Graphite Graphite Graphite Graphite Graphite Graphite Graphite
Locality	Grafton. Mudgee. Undercliff. Undercliff. New England. Fairfield
Date	1888 1888 1890 1894 1895 1895

\* Mineral Resources of New South Wales by E. Pittman, 1901, page 372.

#### CHAPTER V.

# QUALITIES OF GRAPHITES FOR COMMERCIAL PURPOSES.

The application of the graphites varies with their morphologic and physical properties and chemical composition (Proportion of carbon to the ash and conditions of the latter.) Apart from several minor uses, there are three principal applications. 1. For pyrometric purposes, for the manufacture of crucibles. 2. For the manufacture of pencils. 3. For anti-friction compounds and for paints.

For a long time it was held that in the determination of the relative values of the more or less impure graphites, which are mostly used for pyrometric purposes, the quantity of carbon alone was decisive; but practically speaking this is only of importance in so far as graphite as an element is one of the most important ingredients in crucibles, the quantity of which cannot fall below a certain minimum.

Concerning the properties of graphite for pyrometric purposes, C. Bishop,\* an authority on fire clay and similar materials, expresses his views as follows:

- 1. In the application of graphite for pyrometric purposes, that is as an addition to fire clay in crucibles, the quantitative determination of carbon, as well as of its components, is not essential. Neither a larger or smaller quantity of carbon, nor that of the admixture (to a certain degree) is decisive.
- 2. It depends, however, primarily upon the quality of the accessory ingredients, and more particularly upon the proportion of the alumina to the fluxes.
- 3. The quality of the carbon, the greater or lesser incombustibility, is only of secondary importance. In equal or similar total proportions the quality of the carbon is decisive.
- 4. The chemical analysis as a rule is of the utmost importance and is in all cases a sure guide. If the analysis gives equal and compensating values, the practical criterion is the pyrometric test.

<sup>\*</sup> Dinglers polyt. Journal CCIV, 139.

5. Both tests, the analytical as well as the pyrometric, should be made, however, in all cases for the sake of control, even if they are not always necessary. An agreement between the analytical and pyrometric results is the proof of accuracy.

For the manufacture of crucibles, it is essential that the graphite be almost free from all those components which render after the mixture with fire clay the latter less effective in its resistance against high temperatures. In this connection it may be noted that graphite of a lesser value can be effectively replaced by retort coal or coke.

The quantity of carbon does not decide the actual value of a graphite. The application of a graphite for the manufacture of crucibles is dependent upon the higher degree of crystallization, that is, upon the larger and smaller quantity of graphite scales or laminæ.

Ceylon and Mariinskoi graphites are to some extent not only scaly but also highly fibrous (like wood fibre) to columnar, and it is only the scaly varieties which can be used effectively for high grade crucibles. Further, the combustibility, the quantity and quality of the ash are in close relation to each other, and the pyrometric value is determined according to the quality of the accessory constituents, especially the proportion of alumina to the fluxes.

Stingle\* expresses himself as to the qualifications of the graphite as follows:

Graphites for colouring purposes can only be taken from the amorphous and earthy material like the Bohemian and the refined varieties. For the manufacture of crucibles, however, besides the determination of the constituents of the ash and the pyrometric qualities, due regard must also be given to the scaly or lamellar structure of the graphite; because it is a well established fact that such lamellar graphite resists effectively the fire for a longer period than the amorphous varieties; it prevents the cracking of the crucibles, which might be explained by the easy dislocation of the single laminæ or scales, caused by sudden changes in temperature without disturbing the coherence of the material.

As to the application of the dense, earthy or amorphous graphite to the manufacture of crucibles, Weinschenk† remarks that many of these are used at present in the steel industry, both for the manufacture of crucibles and for foundry facings. But he

<sup>\*</sup> Berliner Berichte, 1873, page 391.

<sup>†</sup> Donath, der Graphit, 1898.

points out that crucibles made of fire clay and dense, earthy graphite do not possess by any means the valuable qualities in the same measure as those made of fire clay and scaly graphite. But as the greater part of crucibles, considering the very high temperatures to which they are exposed in the manufacture of steel, would not stand more than two or three charges any way, and as the difference in price between the dense amorphous and the scaly graphite is very great (the latter costs from 5 to 10 times the former), it is understood that in many of the crucibles nowadays used in the manufacture of steel the dense and amorphous varieties are used. Generally speaking, crucibles made from this material do not stand more than one, sometimes two charges.

The difference which exists in the quality of crucibles, in the application of a slaty, scaly and in that of an earthy graphite is, according to Weinschenk, a very wide one: crucibles made with the latter do not possess the tenth part of resistance possessed by those made with the former. An explanation of the cause of this difference is not difficult to find: the scaly graphite, which is intimately mixed with clay, forms in the latter a more or less coherent skeleton which imparts to the clay a high degree of solidity. further the graphite scales along their planes very easily slip past each other, it is evident that the crucible possesses also a high degree of elasticity, which renders it highly resistant to sudden changes in temperature. In the case of the employment of earthy, amorphous graphite, however, the latter is more evenly mixed with the fire clay; the single graphite individuals are not in close contact with each other, and the crucible possesses only a small resistance against cracking. This explains the high prices of the finer qualities of the scaly graphite. The product of the Island of Ceylon, which enters the market mostly as natural graphite, and shows in this state a great purity, represents, therefore, considering the enormous masses in which the graphite is mined there, extraordinary high values. The extraction of the scaly graphite from the rocks, which contain from 5 to 15 per cent. of this quality, may be considered still profitable, as the mines in the Passau district and in the United States demonstrate. As to the structure of Cevlon and Passau graphite, it must be mentioned that besides the long scaly, lamellar varieties, a columnar form with a parallel or radiated arrangement occurs, the leaves and columns being often banded and broken. The deposits near Passau are mined almost exclusively for the purpose of the manufacture of crucibles.

contents of graphite in the numerous ore lenses is very variable, as is also the size of the scales; this variation is greater the more closely one approaches the granite boundary. In the Passau graphite the refractory graphite scales are separated from the adhering earthy graphite or gangue by a certain milling process, and a refined graphite, which is largely used for crucibles, consists of 62.8 per cent. carbon, 33.8 per cent. ash, and 3.4 per cent. volatile matter. By this milling, according to H. Putz, up to 27.7 per cent. graphite scales with 26.7 per cent. ash, out of the total run of mine, can be obtained; and by a certain treatment with crude petroleum, 63.8 per cent. graphite scales, with 17.4 per cent. ash, can be extracted, the degree of extraction being generally dependent upon the more or less loose conditions of the graphite mass, caused by weathering. The fire resisting qualities of the refined Passau graphite, and of the graphites generally used for the manufacture of crucibles, increase, according to Putz, with the contents of ash. They are dependent upon the silica skeleton of the graphite scales, the former remains in the form of the latter in the graphite ash.

In judging of a graphite for crucible purposes, H. Putz\* lays greatest stress upon the difficult combustibility of graphite; while he regards the possibility of the ash constituents as of only secondary importance. Comparative tests of various kinds of graphite of the same size of grain gave the following results in regard to combustibility of carbon:—

<sup>\*</sup> Jahresberichte des naturhistorischen Vereins Passau, 1886.

TABLE 20.

OF GRAPH	HTES	FOR	CO	MMI	ERCI	AD	P
Number of hrs. before obtaining constant weight of ash.		ಣ	œ	14	10	ec	
e Graph- ter, cal- arbon.	4 bour	100.	89.68	90.94	88.24	:	
gnition of the olatile mati veights of c	3 hour 4 hour	100.	93.85	80 83	87 50	100.00	
Percentage loss upon ignition of the Graphite freed from volatile matter, calculated on equal weights of carbon.	1 hour 2 hour	96.16	85.50	71.00	80.58	97.50	
Perce <b>nta</b> ite fre culated	1 hour	89.74	71.85	61.58	69.41	94.50	
Ash		2.5	0.6	32.5	15.00	Trace	
Carbon		97.5	83.5	62.5	85.00	100.00	
Volatile Matter.		0.0	7.5	5.0	0.0	0.0	
No.		1		3	4	5	

No. 1. Ceylon graphite of excellent quality. 2. Ceylon best quality. 3. Ceylon ordinary quality. 4. Bavarian purified. 5. Bavarian, freed from ash by means of hydrofluoric acid.

It thus appears that the Bavarian graphite is more refractory than the Ceylon graphite. It is noticeable that the combustibility of the carbon is considerably diminished by the ash present, so that in judging of the quality of crucibles, the proportion of carbon can by no means serve as the only guide.

In contrast with the Passau ore, the graphites of southern Bohemia are mostly dense, earthy or amorphous, more rarely fine, scaly, and the ore bodies are here larger and generally holding out over a longer distance. These dense varieties are useful only when they show a content of from 45 to 50 per cent. carbon. Occasionally a carbon content of 70 per cent. is met with. A higher degree of 80 to 85 per cent. is only found in the so-called "fat" graphite in the mines of Schwarzenberg; this quality is a soft, friable, exceedingly fine, scaly variety, which represents the most valuable material at present obtainable for the manufacture of pencils. The thickness of the ore body, which produces this graphite, and which is enclosed by graphite ore bodies of inferior quality, has proven to be considerable; its extension along the strike is very large, so that this particular occurrence is at present one of the most valuable graphite deposits so far discovered.

Of the many qualities of graphite, which makes it a mineral of such high commercial value, may be named; the metalliferous. pure black streak and its very low degree of hardness, both of which combined facilitate the application of the same to the manufacture of drawing pencils. But the number of graphite deposits. which furnish a material applicable for the purpose under consideration, is very small, indeed a great number of natural occurrences of known pure quality cannot be used at all, while only a very few of the refined varieties possess the qualities so essential for the production of good pencils. Good pencil graphite must not only possess in its natural state great purity, but also special physical qualities. The most important of the latter is the accuate size of the laminæ. Very dense graphites have, of course, a high colouring power, but they produce a dull, pale streak on paper and the powder does not adhere so well to the latter. Coarse scaly graphites, on the other hand, are not at all suitable for the manufacture of pencils, because the scales or laminæ exhibit on their surface a more or less unctuous condition, and for this reason

glide over paper without losing more than a few very fine minute seales of a highly metallic lustre, but without leaving a mark. If, therefore, the material is not finely pulverized and perfectly homogeneous, every scale present in this mass will cause a slipping of the pencil.

On the other hand graphite possesses such a small degree of brittleness that it resists effectively pressure and blows and can therefore be broken up only with great difficulty. In this connection it may be said that it is far easier to break a hundredfold harder kernel of diamond than a small scale or leaf of graphite. For the manufacture of pencils, therefore, only quite homogeneous, fine, scaly, lamellar varieties can be used. The coarse crystalline graphites which occur quite abundantly in large masses and in many localities over the globe, as well as most of the earthy, dense or amorphous qualities, are unsuitable for this purpose.

Tests have been made, based on a certain reaction of the coarse crystalline graphite, with a view of utilizing the latter, which is less costly for the manufacture of pencils. As already mentioned on page 12, if coarse scaly graphite is treated with nitric acid and then heated, it becomes flatulent and produces worm-like forms, which are called, according to their inventor, Brodies' graphite. These forms are in volume one hundred times larger than the original graphite and are composed of very minute graphite particles and fine compact graphite lamellæ. The latter, however, contain more or less some of the larger scales, and as they must be reduced to the proper size before the whole can be used, it is evident that the graphite produced in this way, although of very pure quality, is not adapted for the purpose under consideration.

According to Weinschenk, really good pencils are made only with the natural, fine scaly occurrences, which are the more valuable the purer and more homogeneous their condition is. The high price, generally paid for the latter, is not an item of great importance, as the quantity used is so very small in comparison to that manufactured into other articles, that the total manufacture of pencils in the world to-day does not absorb 4 per cent. of the total production.

Knapp\* gives his views regarding the application of graphite as follows:—

Graphite applicable for the manufacture of pencils occurs

<sup>\*</sup> Percy-Knapp, Metallurgy, Vol. I, page 230.

most rarely; the finest quality for this purpose is undoubtedly the graphite of Borrowdale in Cumberland, which for this reason is so highly priced. The value of this graphite is, however, not based upon its purity, but upon the grain and structure; for the very pure graphite of Ceylon is not fit for the manufacture of pencils and is much lower in price. In the manufacture of pencils only very fine granular, friable, more or less earthy graphite can be used, while the scaly, slaty graphite of a micaceous structure is the only kind which is applicable to the manufacture of crucibles.

Kretschmer finds that only the fine scaly natural varieties of graphite are most suitable for the manufacture of pencils, while the more common kinds, which can be found in a great number of places, that is the large scaly, as well as earthy varieties, are unsuitable for this purpose. The purer the material, the higher is in this case its value. The mines at Borrowdale, Cumberland, being exhausted, and the Mariinskoi mines in Siberia being idle on account of difficulties with the Russian Government, the graphite mines near Schwarzbach, Bohemia, are the only sources from which at present the factories in Nuremberg, Vienna, as well as France and England can draw their supply of suitable natural graphite.

The very fine scaly graphite of the mines of Borrowdale furnished at one time the material for the best English pencils; only the refuse of these mines was occasionally used for crucibles. Even higher in value for the purpose of manufacture of pencils, is the graphite from the Mariinskoi mine (Alibert graphite), which is for the greater part pure granular and fine scaly; there occur also beautiful fibrous, also columnar and vein-like aggregates.

The Ticonderoga graphite is sometimes of such natural purity that it is advantageously used as a lubricant for all kinds of machinery and apparatus, especially high speed engines, and compared with lubricants manufactured of other material, it is claimed that a saving of from 30 to 40 per cent. can be made. Many graphites of commerce are unsuitable for this purpose, on account of their inclusions of quartz and silicates.

In the manufacture of good grades of pencils, lubricants, electrical supplies and crucibles, it is essential that a nearly pure graphite be used. For paints and foundry facings, purity is not so essential, and therefore the amorphous varieties can be used with as good results as are obtained with the crystalline. The uses of graphite as a lubricant have been constantly increasing, and now

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there are many forms of graphite lubricants on the market. Many reasons are advanced for the more general application of graphite as a lubricant. There is the fact that the graphite permanently fills up all the minute irregularities or roughness on metal surfaces, thus making them absolutely smooth and even; It also reduces frictional resistance; is not crushed or squeezed out by a great pressure; prevents sizing and cutting; is unaffected by any degree of heat, that would be attainable in a cylinder or bearing; is not decomposed by the action of anything to which it would be subjected, and it does not corrode metals, with which it comes in contact. It must, however, be an absolutely pure graphite, without any gritty matter or iron oxide in it.

#### QUALITIES OF CANADIAN GRAPHITES.

As to the adaptability of the Canadian graphite for pyrometric purposes and for the manufacture of pencils, it must be said that the highly crystalline character and the pureness of some varieties admits of its application for both purposes. Prof. Dr. Bischof of Wiesbaden, Germany, the well known authority on fire resisting materials, has made a series of investigations with parcels of ore from the Black Donald mine, Renfrew county, and has compared them with the best Ceylon and Passau graphite, used generally in the manufacture of crucibles. The results were as follows:—

Sample No. 1.—The structure is massive, dense and compact, made up of fine minute laminæ. The color is dark steel gray, and the lustre of a freshly fractured surface, submetallic, that of worn surfaces, bright metallic. This graphite was free from organic impurities. Heated in the closed tube, it gave a little water, but not more than sufficient to form a film. Treated with hydrochloric acid, it formed a black powder more amorphous than scaly.

Sample No. 2.—Pure scaly graphite obtained by sifting powdered lump graphite. The structure of the latter was massive, made up of coarse and thick laminæ presenting a kind of radiated arrangement. The color is dark steel gray with a highly metallic lustre. A portion of the foreign mineral matter in the original lump graphite consisted of calcite. When heated in the closed tube it gave off water and carbonic acid gas.

Sample No. 3.—Flake graphite, consisting of fine scaly and dense graphite. This specimen contained very thin seams of foreign mineral matter.

The results of the pyrometric tests were:-

Sample No. 2, according to two determinations, was at least equal to the best Ceylon—then follows Sample No. 3 which, in its refractory qualities, was of a higher grade than Passau graphite, but considerably lower than Ceylon graphite. No. 1 was only a little above Passau ore.

A series of investigations of Canadian and Ceylon graphite for the purpose of comparison in their relative value as to combustibility have been conducted by Dr. C. Hoffmann\* and the results obtained are given briefly in the following:—

"As to the methods employed for the determination of the combustibility, it must be said that this depends upon the difference in loss sustained by the specimen under trial as compared with that of the specimen of Ceylon graphite employed as the standard, when ignited under precisely identical conditions.

"In the selection of the various graphites it was sought to bring them into the nearest possible accordance as regarded the percentage of ash, for which reason the purest obtainable specimens were in all cases chosen. The percentage of ash in the graphite employed in these experiments was determined after ignition and the necessary corrections were made for the same in calculating the results. The samples were all ignited previous to use, in order to expel volatile matter, thereby insuring that loss from this source should not be attributed to loss by carbon. The graphite was, in all instances, reduced to the same state of mechanical division.

<sup>\*</sup> Report Geolog. Survey of Canada, 1876-77, page 489.

TABLE 21.

	Spec		Per Cent.		Relativ	Relative Combustibility.	ility.
Locality	Gravity	Volatile Matter	Carbon	Ash	Method 1	Method 1 Method 2	Method 3
—Ceylon vein graphite variety foliated	2.2664	0.108	629.66	0.213	1.00	1.00	1.00
Ceylon vein graphite variety columnar.	2.2546	0.000	98.817	0.283	1.02	8.5	1.0
Ceylon vein graphite variety ionated	2.2484 $2.2671$	0.301	99.284	0.050	1.35	1.81	1.25
Canada, Buckingham, disseminated scaly		:			:	1.05	1.05
anada, Buckingham, disseminated scaly.	:	:	:	:	1.01	1.02	1.0
foliated	2 2689	0.178	99 675	0.147	66.0	1.01	1.00
8—Canada, Buckingham, vein graphite variety	2.2679	0.594	97.626	1.780	1.00	1 02	1.01
9—Canada, Grenville, vein graphite variety toh- ated	2.2714	0.109	99.815	920 0	1.01	1 03	1.02
vein graphite	2.2659	0.108	99.757	0 135	1.12	1.12	1.12
N.Y., vein	2.2599	1.191	96 656	2.153	1 03	1 00	1.01
12—N. S. Liconderoga, N. Y. vein grachite variety foliated	2.2647	0.818	97, 422	1.760	1 01	1.00	1.00

"In selecting the standard the choice lay between 1 and 3, for the reason, however, that the latter is understood to be the most expensive, it was concluded that it would be scarcely likely to meet with such an extensive application in the manufacture of crucibles as the former to which, in consequence, the preference was given. The figures given under method 1 and 2 are in both instances the mean of two closely concordant determinations: they represent the amounts of graphite burnt off as compared with 1.00 of that of the graphite employed as standard (Ceylon 1) when ignited under precisely identical conditions. It may be added that in appearance the Ceylon graphite was, without exception, undistinguishable from the Canadian, the exception being 4, the structure of which entirely differed from that of any of the Canadian specimens, the only one of the latter at all approaching it in this respect being 10, and this only in parts, the remainder of the structure being much coarser. As will be seen these two specimens were the most combustible of the Cevlon and Canadian graphite. There appears to be some, if indeed it may not be said, a close connection between the combustibility of the graphite and its resistance to mechanical division (pulverisation); those most difficult to pulverize being the least combustible.

"From these experiments it will be seen that in respect to combustibility the Canadian graphite may claim perfect equality with that of Ceylon; and that, consequently, apart from any consideration of the proportion and nature of the associated foreign matter, it is in no wise inferior to the latter as a material for the manufacture of crucibles.

"As to the disseminated material, it must be mentioned that the same is apt to contain more or less carbonate of lime and oxide of iron, and that these impurities must be eliminated either by dressing or refining, either wholly or for the greatest part at any rate, in order to render the graphite useful for the above purposes. In the case of samples 5 and 6, the pulverized material was treated with hydrochloric acid, with the aid of heat, and the resulting solution contained large quantities of lime and iron, while the percentage of mpurities was insignificant, leaving the graphite with a very small amount of ash, and this in no wise prejudicial to its application for the purpose here under consideration. That the graphite from this source in itself compares favourably with that of Ceylon, will be seen from the above table."

As a further proof of the excellence of Canadian graphite in

the manufacture of crucibles, it may be mentioned that some two years ago 65 tons of flake ore from the Calumet (Grenville) properties were sent to and refined by the Globe Refining Co. of Jersey City, N.Y., and the product, some 30 tons, sold to some of the largest crucible makers in the United States, England and Germany, who pronounced it equal to the best Ceylon graphite; these sales resulted in numerous inquiries from the purchasers and from other users.

As to the employment of Canadian graphite for pencils, investigations have shown that several varieties from Buckingham and other localities are well adapted for the manufacture of pencils of good quality. Some of these varieties not only possess in their natural state great purity, but also special physical qualities, which make them very suitable for this purpose. The writer has made a number of experiments with different classes of ore at a large pencil factory in Nuremberg, Germany, and these tests show, without doubt, that certain varieties on account of their exceedingly fine crystallization, if properly selected, produce pencils which compare very favourably with some good grades made from the Bohemian graphite.

The graphite so employed by the writer contained carbon varying between 85 per cent. and 97 per cent., the carbonate of calcium and other soluble constituents having been extracted by the application of hydrochloric acid.

As a lubricant, Canadian flake graphite is so well known on the market, that it is not necessary here to dwell on this subject at length; it suffices to state that its purity and the uniformity of the flake, as now produced by the Canadian mill owners, give it first rank amongst the lubricating graphites now offered in such large variety; this is also shown by the fact that the Canadian article is amongst those which command at present the highest prices.

#### CHAPTER VI.

# DETERMINATION OF THE VALUES OF GRAPHITES.

In most cases the value of a graphite depends principally upon the quantity of carbon, so that the determination of the latter may be considered sufficient; but if the graphite is used for more specific purposes, it may be necessary to determine not only the quantity of ash, but also those constituents in the latter, which act as fluxes, that is, iron oxide, alkalies and alkali earths. Further, for the application of graphite to the manufacture of crucibles for steel melting processes, it is essential that the graphite contains very little sulphur, and the quantity, if any present, must be determined also.

Many of the cheaper grades of commercial graphite are adulterated with coal dust, and such adulteration may be detected by the following simple test. About a quarter of a gram of graphite powder is mixed in a test tube with 15 cc. of acctone, and the whole is then allowed to stand for 10 or 15 minutes. It will then be observed that pure graphite settles clear, leaving the liquid colorless, coke dust imparts a gray color to the solution and remains in suspension a long time; anthracite dust imparts a faint brown color and settles more rapidly, while soft coal dust imparts a deep brown color to the acetone. Equal parts of glacial acetic acid and sulphuric answer as well as acetone for such a test.

Molybdenite and graphite closely resemble each other, especially when they are disseminated as small flakes in a rock. They are both soft, and have a greasy feel, and they occur in various ways from minute scales to large foliated masses. The larger flakes can readily be told apart by the colour, as molybdenite is bluish lead-grey, while graphite is dark steel-gray to dull black. However, when either occurs in small specks or particles in a rock, the colour cannot always be judged. One of the simplest tests is to mix some of the rock powder containing the mineral with potassium nitrate on a thin piece of platinum or tin and apply a flame beneath. Graphite will deflagrate, that is,

will burn with miniature explosions like grains of gunpowder, whereas molybdenite will remain unaffected. Very small amonnts of graphite in a rock can be detected by this method.

### Determination of Carbon by Combustion.

Although there are a number of simple and accurate methods for the determination of carbon, there are certain difficulties in the case of graphite, because of the relatively high temperature, at which the combustion of carbon takes place, and this only under abundant access of air or oxygen; the latter must be employed in case of quick determinations.

If the carbon is to be calculated out of the loss in weight of the material after the combustion is finished, it is of importance to observe the following rules:—

- 1. In the presence of chemically combined hygroscopic water the graphite must be heated to a temperature of 150° C. and the loss, so determined, must be deducted from the total loss in weight after incineration.
- 2. Graphite of the crystalline formation may contain carbonate of calcium. During the combustion process in the crucible, the carbonic acid gas is expelled and consequently after incineration the contents of carbon will appear too high. It is hence necessary to determine the carbonic acid and deduct the weight from the total obtained by oxidation.
- 3. If iron pyrites is present, the sulphur disappears during the process of burning, while the iron combines with oxygen to iron oxide. As 240 parts of iron pyrite when heated leave 160 parts of iron oxide, it is evident that if this loss in weight is not taken into consideration, the contents of carbon will appear somewhat high.

The presence of hydrogen, oxygen and nitrogen in graphite has also an influence upon the final result, but as they are generally present in very limited quantities, for practical purposes, the loss in weight from this source may be neglected.

4. Many graphites leave a fusible ash, this ash envelopes the fine particles of carbon, prevents their direct contact with oxygen, and consequently their free complete combustion. The burning of these graphites gives inaccurate results, and other methods for the determination of the carbon must be resorted to.

The total mineral constituents of a graphite is indirectly found by deducting from 100 the carbon, moisture and chemically combined water, expressed in per cents. If a direct determination is required it suffices, for many kinds of graphite, to place a small quantity (about 0.5 grams) of the finely powdered substance in a platinum crucible, and expose the same, with free access of air to the long continued and strong heat of a Bunsen or Maste gasburner. F. Stolba\* recommends a platinum crucible, provided with a projecting perforated lid, the round hole in which is 5 millimeters in diameter. The crucible is fixed in an inclined position, and the cover is so placed that about one quarter of the opening is left uncovered. The combustion of the carbon is facilitated by exposing a fresh surface of the graphite, by turning the crucible round occasionally or stirring the contents with a platinum wire. As the operation requires from 3 to 4 hours for its completion, and as the weight of the platinum crucible may be affected by so prolonged a heating, the crucible must be weighed again. If a muffle is available, the combustion of the carbon may also be accomplished in a platinum dish, placed in the muffle. heated to redness. This method, which permits of the incineration of large quantities of graphite, is particularly to be recommended, when the ash is to be further analysed.

If the graphite contains calcium carbonate, the carbonic acid is naturally expelled during ignition; this can be replaced by moistening the ash repeatedly with a concentrated solution of ammonium carbonate, dried and gently heated. But a complete agreement between the quantity of the mineral constituents directly determined, and that directly found, cannot always be expected even after the treatment with ammonium carbonate e.g. when the graphite contains iron sulphide or ferric hydro-oxide.

Stolba† proposes to burn off the graphite in the presence of fine granulated silver (obtained by reduction of silver chloride). He takes 2 grams of graphite, adds one gram of the finely granulated silver and burns the whole in a small flat platinum dish of 5-6 centimeters diameter over a Bunsen burner. In order to facilitate the circulation of the air the platinum cover is placed in an inclined position over the vessel. After 15 minutes the incineration is interrupted, and, after cooling off, the mass is thoroughly stirred and mixed, again subjected to a red heat until the

<sup>\*</sup>Dinglers Polyt. Journal CXCVIII 213 and Fresenius, page 718. † Chem. Centralblatt, 1888, 1-301, also Donath, der Graphit, page 168.

graphite has all disappeared. The addition of the finely granulated silver facilitates the combustion of the graphite. During the process of burning, care must be taken that the silver does not melt, as otherwise the platinum vessel would be lost.

Mr. F. S. Hyde\* gives his views regarding the combustion and fusion method as follows:—

"Exclusive of the more elaborate methods by combustion and subsequent weighing as carbon dioxide, the choice lies between direct oxidation by blast (or muffle) and the method by fusion with caustic potash. In the one, the determination is made by loss through oxidation; in the other, the graphite is obtained and weighed in the purified state. Both of these methods have advantages in their simplicity; but for manufacturing purposes, the blast is superior for ascertaining the refractory properties and suitability of certain graphites for metallurgical purposes.

"In the method by blast, allowance should be made for free and combined moisture and sulphurous and organie volatile matter. From 0.5 to 1.0 gram of the pulverized substance is weighed in a platinum crucible, which is then placed in a vertical position, covered and subjected to a red heat for one minute over a Bunsen flame. On cooling to a low red heat, the crueible is momentarily uncovered and rotated to oxidize any traces of sulphur, which, when present, usually passes off with slight fumes and odor of sulphur dioxide. Should sulphur be present in larger amounts, proceed cautiously, reheating and cooling until no more fumes or odor are noticeable. Mere heating under cover is not sufficient to drive out sulphur from pyrites, oxidation at a low red heat seems essential. Tests have shown that such procedure gives eoncordant results without appreciable oxidation of graphite. Having estimated all matter volatile at a low red heat, the crucible and cover are inclined and subjected to a direct continuous blast maintained by compressed air. The lamp should be regulated to give a clean blue flame without unnecessary noise, and the graphite should be stirred oceasionally with a stout platinum wire to facilitate oxidation. The operation may require from one to five hours, according to the nature of the graphite, but usually not more than two and one half hours."

The most accurate method for the determination of earbon is the combustion in a current of oxygen. This method has been

<sup>\*</sup> Mineral Industry for 1900, page 380.

used by the writer on Canadian, American and Bohemian ore, and has given excellent satisfaction. The procedure is as follows:

- 1. Reduce the graphite to a fine powder so that it can pass an 80-mesh sieve. From 0.5 to 1.0 gram of the pulverized substance is weighed into a porcelain crucible, which is then placed in a vertical position, covered and subjected to a low red heat for one to two minutes over a Bunsen flame. Or dry a sample in an air oven, in which a temperature of 150° is maintained. Determine loss in weight.
- 2. Determination of carbon:—One gram of the originally pulverized sample is placed in a Rose porcelain crucible and the whole weighed, then subjected to a continued red heat over a Bunsen flame; see fig. 13, a current of oxygen is supplied through

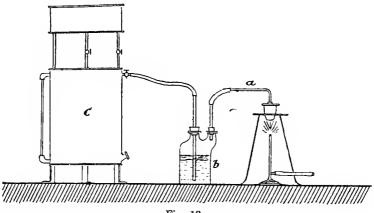
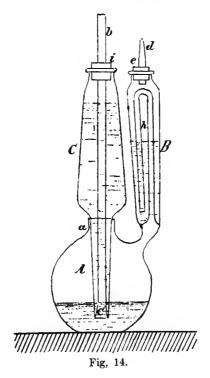


Fig. 13.

tube a, made of French clay, and inserted in the cover through an opening of 5 millimeters in its centre. This tube is connected indirectly with a gasholder c, and in order to control the flow of gas, a wash bottle b is inserted in the manner illustrated, which, by the number of bubbles rising through the water, indicates the velocity of the current. During combustion, change the surface of the graphite repeatedly by stirring with a platinum wire. Continue the heating of crucible until the color becomes light gray, which is a sign that all graphite is burned. The operation requires from one to two hours, according to the nature of the graphite. Take weight of crucible and ash. The loss in weight represents: Volatile matter as determined before; carbon and, if carbonate of calcium is present, also carbonic acid gas.

3. Determination of carbonic acid gas in graphite ores:—In order to determine the quantity of carbon dioxide contained in a carbonate, several methods may be employed, one of which consists in the determination of the loss of weight, which the ore undergoes when an acid is added. The following is a description by Fresenius of the apparatus constructed by Geissler, usually employed for this purpose. The apparatus, the construction of which is shown in Fig. 14, consists of three parts A, B and C. C is



ground into the neck of A, so that it may close air tight and yet admit of its being readily removed for the purpose of filling and emptying A. b c is a glass tube, open at both ends and ground water tight into C at the lower end a; it is kept in the proper position by means of the moveable cork i. The cork e must close air tight, and so must the tube d in the cork. The graphite ore to be decomposed is put into A, water is added to the extent indicated in the fig., and the substance shaken towards the side of the flask. C is now filled nearly to the top with dilute nitric

or hydrochloric acid, with the aid of a pipette, after having previously moved the cork i upwards without raising b. The cork is then again turned down. C is again inserted into A, B somewhat more than half filled with concentrated sulphuric acid, and b closed at the top, by placing over it a small piece of caoutchouc tubing with a glass rod fitted into the other end. After weighing the apparatus, the decomposition is effected by opening b a little, and thus causing air to pass from b into the sulphuric acid, where it is dried. It leaves the apparatus through b. When the decomposition is effected, b is gently heated, the stopper from b removed, and the carbon dioxide still present sucked out at b. The apparatus when cold is again weighed and the difference between the two weighings will be that of the carbon dioxide expelled.

Deduct the latter and the weight of the volatile matter as determined under (1) from the total loss as obtained under (2), and the result is the quantity of carbon contained in the graphite in per cent.

4. Preparation of oxygen:—A simple method for preparing oxygen consists in heating potassium chlorate, commonly called chlorate of potash (KClO<sub>3</sub>). This salt loses the whole (39.14% of its weight) of its oxygen, leaving potassium chloride:

The temperature has to be raised much above the melting point of the salt to about 350° before the evolution of the gas begins; and after a certain time has elapsed, the fused mass becomes thick, owing to the formation of potassium perchlorate. When more strongly heated the perchlorate also decomposes into potassium chloride and oxygen.

In order to obtain the evolution of oxygen at a lower temperature, a small quantity of manganese dioxide is generally mixed with the powdered chlorate; the gas is liberated then at 200° C, before the salt fuses, and thus the preparation of the gas is greatly facilitated. The manganese dioxide is found mixed with the potassium chloride in the residue wholly unaltered. The apparatus most commonly used for the preparation of oxygen is illustrated in Fig. 15. The mixture of potassium chlorate dioxide is heated in a thick copper vessel A, provided with a wide tube connected with the wash bottle B, containing caustic soda, for the purpose of absorbing traces of chlorine gas, which are generally

evolved owing to the presence of dust and other organic matter in the mixture. The gas is collected in gasholders c as illustrated in Fig. 13.

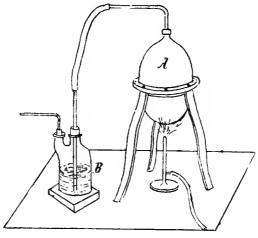


Fig. 15.

Note.—It not unfrequently happens that the economical black oxide of manganese may be accidentally mixed or adulterated with carbon (pounded coal) and this impure material, when mixed with chlorate of potash and heated, ignites, giving rise to even fatal explosions. Hence, care should be taken to try any new sample on a small scale beforehand by heating it with chlorate of potash in a test tube.

A. G. Stillwell,\* while making a determination of carbon in steel by the absorption method, observed that this method, with some minor changes and additions, might be made to apply to the determination of graphite in ores. After some experimenting, the following method was devised, and has been used for some time with great success.

<sup>\*</sup> Journal Chemical Industry, Vol. XXI, 1902, page 759.

The following enumeration of apparatus employed will serve to simplify the description. See Fig. 16.

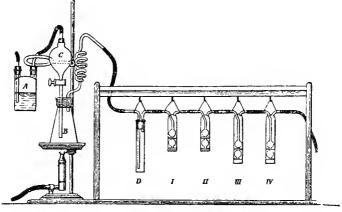


Fig. 16.

- A—Guard bottle containing caustic potash in solution.
- B—Generator flask. (An Erlenmeyer, with mouth 1½ in. in diameter.)
- C—Tube for running in sulphuric acid.
- D—Empty test tube to catch water carried over.
- I and II—Guard bulbs, sulphuric acid (concentrated).
- III—Caustic potash bulb for absorbing  ${\rm CO_2}$  (KOH, 1.27 sp. gr.)
- IV—Bulb for sulphuric acid (concentrated).

The following amounts of the finely ground sample (80 mesh) will be found most satisfactory for use:—

For ores of over 25 per cent. carbon, use 0.25 gram.; for ores running between 15 and 25 per cent. of carbon, use 0.5 gram.; for ores running below 15 per cent. of carbon, use 1 gram.

The material is weighed on balanced glasses and brushed into a deep platinum or porcelain crucible. Heat is applied to dull redness for a few minutes to drive off any organic matter that may be present. The material is now transferred to a small beaker, and dilute HCl (I—I) added, and heated to boiling to decompose any carbonate and expel CO<sub>2</sub>. The solution is now filtered through a porcelain Gooch crucible, using ignited asbestos as filtering medium

and washed two or three times with hot water until the HCl is all gone. The crucible, with its contents, is now placed in the flask B, and 15 c.c. of a saturated solution of chromic acid added.

The apparatus is connected (after weighing III and IV) and all air is drawn out by means of suction. Seventy-five c.c. of  $\mathrm{H_2SO_4}$  (concentrated) are now put into bulb of tube C and run in very slowly, gently rotating flask D at the same time, so as not to have a violent evolution of gas. When the  $\mathrm{H_2SO_4}$  is all in, close stop-cock and heat flask with small flame till heavy fumes fill the flask. Now turn flame very low, open stop-cock so that one bubble per second passes through solution in A, and continue aspiration for half an hour. Disconnect and weigh III and IV. The increase in weight represents  $\mathrm{CO_2}$  which, multiplied by 0.2727, gives the amount of carbon.

The whole operation may be run in two hours, with no attention from the operator during the last hour.

If no organic matter or carbonates be present, the two steps relating to them may be dispensed with, and the weighed material brushed directly into flask B. In this case the operation requires one hour.

By running a number of blank tests, i.e., using chromic and sulphuric acids, it was found that III and IV increased in weight by 0.0045 grams. This is to be deducted from weight of III and IV, before calculating results.

The method gave, on various ores, the results shown in Table 22.

The first two ores (low grade) were run in thirty-five minutes from first weighing.

Description of Ore.	Amount used	Weight of CO <sub>2</sub>	Carbon			
	Gram.	Gram.	'   %			
Low grade ore	1	0.085	2.31			
Quartz gangue		0.084	2.29			
Low grade ore	. 1	0.1740	4.74			
Quartz gangue		0.1710	4.66			
Containing limestone		1.440	39.27			
		1.445	39.40			
Containing limestone	. 1/4	1.096	29.90			
8	·	1.096	29.90			
Pure Ceylon Graphite	. 1/5	3.615	98.58			
	·		ash 1.42)			

TABLE 22.

Dr. J. T. Donald, of Montreal, recommends the following method which, as a general rule, gives very quick and accurate results: Treat ½ gram of ore in a platinum crucible with 5 c.c. hydrofluoric acid and 2 c.c. sulphuric acid; place crucible on hot plate and evaporate sulphuric acid. This treatment is repeated on low grade ores (containing much gangue); the residue in the crucible is transferred by means of hot water to a beaker and then treated with a little "aqua regia," which latter dissolves the sulphides, iron, alkaline earths, etc. The residue from this treatment is transferred to counterpoised filters and well washed with hot water until free from acid. The filters and contents are then dried in the air oven at 105° C. and weighed.

# DETERMINATION OF CARBON BY FUSION.

In the fusion method, the mineral matter is removed by the action of the molten alkali, while the graphite remains purified and undissolved. The sample should be finely pulverized unless the graphite consists of thinly laminated particles, which can be easily penetrated by the molten alkali. Many samples, however, resist reduction to powder beyond a certain point, and the use of a blast, with free access of air, becomes almost imperative for an accurate determination. The same holds true with certain samples to be tested in the form and size as milled by the manufacturer.

Loewe\* determines the carbon by fusing the graphite with soda potash carbonate, washing the mass with water, boiling the residue with caustic soda and washing the same with hydrochloric acid. The remaining substance is collected on a filter, dried at 100° C., and the purified graphite weighed. By the fusion of the alkali carbonate and boiling of the fused mass with water, the silica is supposed to go into solution. The hydrochloric acid dissolves the liberated metal oxides, but the method cannot give accurate results, because one is never sure whether the final residue is really pure carbon.

Of the methods proposed for rapid determination of carbon in graphite, that of Gintl† may be described. Gintl uses a stout tube of refractory glass 10 to 12 centimeters long and about 1 centimeter wide, sealed at one end and blown out to a small sized bulb. He brings into this tube 0.05 to 0.1 gram of the graphite, dried at 150° to 180°, and added to this 1.5 to 3 grams of pure pulverized

† Ibid.

<sup>\*</sup> Dinglers Polyt. Journal, 137-445.

lead oxide, previously ignited. The whole is then weighed and the lead oxide with the graphite thoroughly mixed by means of a mixing wire; that part of the tube, in which the charge has been placed, is then heated at first over a Bunsen burner, and finally with a blow pipe lamp, until the contents are completely fused and froth is no longer noticeable. According to Gintl this operation is completed in ten minutes. The whole is then allowed to cool, and from the loss in weight, which represents the carbon dioxide, calculate the carbon. Of course, the results obtained by this method are serviceable only when the graphite contains neither water chemically combined or capable of expulsion at 150° to 180° C., nor carbonates, and when all the carbon is oxidized by fusion with the lead oxide.

G. C. Wittstein\* recommends also the well known method of Berthier, used for the determination of the pyrometric value of a substance. One gram of finely powdered graphite is mixed with 25 grams of pulverized oxide of lead, the charge is placed in an unglazed porcelain crucible and then covered with 25 grams of oxide of lead; the whole is heated in a low coal fire. The amount of carbon can be determined from the quantity of reduced lead. Thirty-four parts of the latter correspond to 1 part of carbon according to the equation:

$$2PbO+C=CO_2+2Pb.$$

The method is quite satisfactory if the graphite under investigation is free from metal sulphides, such as pyrites.

Another method is based on the comparison of the reducing power of graphite with the reducing power of pure carbon.† Hence, this method is not adapted for ores containing sulphides, arsenides or antimonides, unless the ore is subject to a preliminary "sweat" or "dead" roast before treatment.

Great care must be exercised that the roasting is complete. The oxides of iron and manganese will also interfere, in which case the oxidizing power of the ore should be carefully determined and correction made for same in final assay.

To obtain the standard reducing power of pure carbon, take 1 gram powdered wood charcoal, 45 grams lead oxide (litharge), 30 grams sodium bicarbonate, 5 grams borax glass. Mix thoroughly, place 20 grams in crucible, add salt, cover and fuse in wing

<sup>\*</sup> Ibid., 216, 45, also Fresenius.

<sup>†</sup> Mines and Minerals, 1898, page 262.

or muffle furnace. After complete fusion, pour in mold, cool and clear resulting lead button of all adhering slag. Weigh button and make correction for ash and hygroscopic moisture in charcoal, which is usually from 2 to 5 per cent. It is well to run duplicates on the above and strike an average.

Having established the charcoal standard, proceed with the graphite. Take 1 gram pulverized ore, 45 grams lead oxide, 30 grams soda, 5 grams borax glass, and if bases, such as iron, etc., in ore, about 15 grams silica. Mix, place in crucible, add salt, cover, fuse and weigh resulting lead button, which will give required percentage of pure graphite in ore.

This method is quick and easy compared with wet determinations. It has only been used on clayey ores, but it seems that if carefully manipulated, results could be obtained on more complex ores, which would be sufficiently accurate for commercial purposes.

F. S. Hyde\* recommends the following method by fusion:-"Use a large silver crucible of about 120 grams weight. From 35 to 40 grams C.P. caustic potash (free from carbonate) are melted in the crucible over a very low Bunsen flame, and fusion maintained at a temperature below an incipient red heat, just enough heat to produce a clear liquid melt. From 0.5 to 1.0 gram of the powdered graphite is carefully introduced on top of the melt (the flame being temporarily removed); the crucible is then covered, the flame replaced, and the contents allowed to simmer quietly, with occasional rotation, for half an hour. Increasing the temperature does not improve matters and may cause loss. The melte after cooling, is dissolved in about 250 c.c. hot distilled water, and filtered by suction on a weighed filter (preferably Schleicher and Schull's No. 590, 11 cm.), which has previously been treated with 1:10 caustic potash, and then with 1:4 hot dilute hydrochloric acid. This washing with alkali and acid is essential, and is preparatory to the subsequent filtration with the same chemicals. For example, one filter taken directly from the package weighed 0.8123 gram, and after treatment 0.7913 gram, a loss of 0.0210 gram, equivalent to 4 per cent. on 0.5 gram graphite taken for assay. After collecting and washing the graphite on the filter, the iron oxide is dissolved with hot 1:4 hydrochloric acid, and may be determined separately in the acid solution. The filter, containing the purified graphite, is thoroughly washed with hot water and dried in the air bath at 70° C.

<sup>\*</sup> Min. Ind., 1900, page 381.

"The results with the blast are remarkably concordant, due, no doubt, in a large measure to the fact that the operation is performed in a single piece of apparatus; whereas, in the fusion method, there is the liability of loss through lightness and oxidation of particles, from overflow from the subsequent treatment in casserole and on suction filter, and even from the charring of weighed filter if dried above 70° C. Filter paper which has been washed alternately with acid and alkaline solutions of moderate strength becomes exceptionally tender, and will hardly withstand prolonged heating beyond 70° C. Asbestos fibre would require similar treatment before use, and even a Gooch filter is not so convenient or simple as good paper, supported with a washed linen cone, for obtaining graphite by direct weight. Furthermore, it is not always possible to judge when the action of the molten alkali is complete. For instance, with the flake product 'B' (Table 23, p. 143), the purified graphite obtained by fusion showed only 98.26% graphite from the blast, and the presence of ash was very evident to the eye. indicating that the molten alkali had not entirely penetrated the original substance. Another sample (part of 'A,' consisting of fine powder) gave after treatment by fusion a product 99.12° pure by blast, the ash being visible. These facts, however, hardly account for the lower results by fusion, which may be due to mechanical losses and possibly to partial oxidation. One of the lowest results was obtained when the sample was weighed in the silver crucible first, and the caustic potash allowed to melt down on top. That the power of penetration of molten caustic is somewhat dependent on its nature and temperature of fusion seems to be indicated by the following experiments, in which C.P. caustic soda was substituted for eaustic potash. Thus the flaky product 'B,' assaying 87% graphite by caustic potash fusion, gave 84.92% with caustic soda. Repeating this experiment on the same material gave 84.26% with caustic soda. As with the caustic potash fusions, the temperature was just sufficient to maintain a liquid melt, and extra precautions were taken to avoid mechanical loss. The product thus obtained by caustic soda was 99.64% pure by blast, the ash being slightly visible.

"A separate lot of similar material assaying 87.54% by blast was then subjected to fusion with caustic soda at an incipient red heat, with the result that only 75.60% graphite was obtained, the loss being evidently due to oxidation in contact with red hot alkali. The purified product yielded 99.74% blast, the ash being slightly

visible. The inference is that caustic potash gives higher results than caustic soda, but the latter yields a product of greater purity.

"Notwithstanding the tendency to low results, the fusion method is one of neatness, and may be employed when the sample contains material fusible at the temperature of the blast, or when facilities for a continuous blast are lacking. Besides, it obviates preliminary determinations for moisture or other volatile matter. Alumina, lime, and magnesia may be determined in the alkaline filtrate, and iron in the acid washings from the graphite residue. For sulphur, a separate determination is preferable, either by treating with aqua regia or by a fusion with an oxidizing agent.

"It may be well to note that silver crucibles after caustic alkali fusion are more easily cleaned than platinum crucibles, which have been coated with fused particles of iron and mineral matter. On the other hand, silver is susceptible to sulphur, and graphite is not always free from this element.

"In commercial transactions, it would be preferable to specify either the blast or the fusion method for general simplicity, convenience and comparisons.

"On account of the tendency to low results by fusion, the following assays on two milled products are submitted for comparison.

# DETERMINATION OF SILICIC ACID, ALUMINUM, IRON, ETC.

To determine the individual mineral constituents so far as the silicic acid, aluminum, iron, etc., are concerned, the ash obtained by the combustion method may be employed, and treated according to the method for the determination of silica, or the graphite itself may be decomposed by some other method. Wittstein\* recommends for this purpose the following method:—

Mix about 1 gram of the finely powdered graphite with about 3 grams sodium potassium carbonate in a platinum crucible, place upon the surface of the mixture about 1 gram potassium hydroxide and slowly heat to redness. From time to time break the crust formed during the fusion with a stout platinum wire. After half an hour fusion, allow to cool, maderate the mass with water, heat for fifteen minutes to boiling, filter and wash the residue. Treat the contents of the filter together with the filter ash with hydrochloric acid of spec. gr. 1.12, and after digesting for half an hour,

<sup>\*</sup> Zeitschr. f. analyt. Chemie XIV, 395.

TABLE 23.

add water, filter off from the insoluble residue of carbon, unite the hydrochloric acid solution so obtained with the alkaline liquid first obtained, and add hydrochloric acid in excess; then evaporate to dryness on a water bath, separate the silicic acid and in the hydrochloric acid filtrate determine the bases. In order to make certain that the carbon filtered off contains no mineral constituents, it is burnt. It is of no advantage to weigh this carbon, as it does not represent the entire quantity of carbon present, but only about four-fifths.

# CHAPTER VII.

# STATISTIC OF PRODUCTION, EXPORTS, IMPORTS AND PRICES.

In order to enable a better study of the economic conditions of the graphite industry, all available statistics regarding the production and exports of the different countries, and consumption of graphite by the different branches of manufacture, have been compiled in this chapter. These statistics have been obtained partly by direct application to the various governments, partly from the "Mineral Industry," and partly from other authentic sources. It must, however, be understood that the figures of the consumption by the various trade branches lack completeness, owing to the different methods of computation employed by the statisticians of the several countries. Nearly all of the figures received include, for instance, pencils made of colors and pencils made of graphite in one figure; also crucibles made of fire clay or of graphite and clay, and in the figures presented allowances have been made for differences.

#### CANADA.

The following statistics of production, exports and imports are compiled by the Geological Survey. It must be stated, however, that no difference has been made by the statistician between amorphous and crystalline graphite, so that the figures presented include both varieties.

TABLE 24.
ANNUAL PRODUCTION.

Calendar Year	Tons of 2000 lbs.	Value.	Calendar Year	Tons of 2000 lbs.	Value
1886	500	\$4,000	1895	220	\$ 6,150
1887	300	2,400	1896		9,455
1888	150	1,200	1897	436	16,240
1889	242	3,160	1898		13,698
1890	175	5,200	1899	1,130	24,179
1891	260	1,560	1900	1.922	31,040
1892	167	3,763	1901	2.210	38,780
1893	nil.	nil.	1902	1.095	28,300
894*	3	223	1903	728	23,745
1094	J	0	1904	452	11,760

<sup>\*</sup> Exports.

TABLE 25. EXPORTS.

Calendar Year.	Value.	Calendar Year.	Value.
1886	\$3,586	1895	\$4,833
1887	3.017	1896	9,480
1888	1.080	1897	4.325
1889	538	1898	13,098
1890	1,529	1899	22,490
1891	72	1900	46,197
1892	3,952	1901	35.102
1893	38	1902	24,839
1894	223	1903	43,642

1903 Crude	wt. ,235 \$26,230 17,412
	\$43,642
1904 Crude	\$27,085 14,643
	\$41,728

TABLE 26.

IMPORTS OF RAW AND MANUFACTURED GRAPHITE.

Fiscal Year.	Plum-	Manufactures of plumbago.	
Piscai Tear.	bago.	Black-lead	Other Manu- factures.
1880			\$2,738
1881			
1882		25,132	
1883		21,151	2,141
1884	_,	24,002	2,152
1885			2,805
1886			1,408
1887			2,830
1888	. 3,802		
1889		11,852	21,789
1890	. 3,441	10,276	26,605
1891	. 7,217	8,292	26,201
1892		13,560	23,085
1893	. 3,293	16,595	23,051
1894	2,177	17,614	16,686
1895	. 2,586		
1896	. 2.865		
1897	. 1,406	17,863	
1898	1,862		
1899	4,979	21,334	
1900			
1901		25,646	
1902	3,649		

	Duty.		1	
Plumbago, not ground, &c Black-lead	10 p.c 25 "	\$2,870	\$22,559	
1903 Plumbago, ground and manufactures of N.E.S. Crucibles, clay or plum-	25 "			<b>\$</b> 12,493
bago	free.			34,624
Total, 1903		\$2,870	\$22,559	\$47,117
1904 { Plumbago, not ground Plumbago, ground and ma Crucibles and clay	nufacture	ed		\$1,802 12,829 28,773
				\$43,404

#### UNITED STATES.

During 1904 the value of the total production of graphite amounted to \$341,372 as compared with the total value of \$225,554 in 1903, an increase of \$115,818, due principally to the large increase in the Pennsylvania, Wisconsin and Georgia productions. Besides the crystalline and amorphous graphite, there is the artificial graphite, manufactured by the International Acheson Co., of Niagara Falls, N.Y. The value of this artificial product in 1904 was \$217,790. Acheson graphite is largely used in the manufacture of metal protective paints, dry batteries, stove polish, packing and as a lubricant. The electro chemical processes also consume a great deal of it.

The production of crystalline graphite in the United States during 1904 amounted to 5,681,177 pounds, valued at \$238,447, as compared with the production of 4,538,155 pounds valued at \$154,170 in 1903, an increase of 1,143,022 pounds in quantity, and of \$84,277 in value. Most of the crystalline graphite reported was refined, but little having been sold in the crude state. The average price per pound received for the 1904 production was 4.2 cents, which is four-fifths of a cent higher than the average price received per pound in 1903.

The production of amorphous graphite in 1904 amounted to 19,115 short tons, valued at \$102,925 or \$5.38 per ton, an increase of 2,524 tons in quantity, and of \$31,541 in value as compared with the production of 16,591 short tons, valued at \$71,384

or \$4.30 per ton, in 1903. The average price per ton is still very low, and is due to the Georgia production which is sold far below the price received for graphite that is used for the many purposes enumerated above.

The following table shows the annual production of graphite from 1880 to 1904, inclusive.

TABLE 27.

Year.	Quantity Pounds.	Value.
880	622,500	\$49,800
881	400,000	30.000
	425,000	34,000
882	575,000	
883		46,000
884	500,000	35,000
885	327,883	26,231
886	415,525	33,243
887	416,000	34,000
888	400,000	33,000
889		72,662
890		77,500
891	1,559,674	110,000
892	1,398,365	87,902
893	843,103	63,23
894	918,000	64,010
895	644,700	52,582
896	535,858	48,460
897	1,361,706	65,730
898	2,360,000	75,200
899	2,900,732	167,106
900	5,507,855	197,579
901	3,967,612	167,714
902	3,936,824	182,108
903	4,538,155	225,554
904	5,681,177	341,372

The annual importation of graphite into the United States each year far exceeds the domestic production and amounted in 1904 to 12,674 tons valued at \$905,581. This is the lowest importation in quantity since 1897, and, with the exception of 1901, the lowest in value since 1898. Since the statistics of the production of graphite in the United States were first collected, there has been no year in which the value of the imports has not greatly exceeded the value of the domestic production. In the following table are given the quantity and value of the graphite imported into the United States from 1867 to 1904, inclusive.

TABLE 28.

Year ending—	Quantity. Long tons.	Unmanu- factured.  Value.	Manufac- tured. Value.	Total value.
June 30—				
1867	1,356	\$54,131		\$54,13
1868	3,431	149,083		149,08
1869	3,742	351,004		351.00
1870	4.040	269,291	\$833	270,12
1871	2,581	136,200	3,754	139.95
1872	4.819	329.030	3,734	329.03
1873	7,877	548,613		548,61
	5,600	382,591		382,59
1874	2,329	122,050		122,05
1875	$\frac{2,329}{2,530}$	150,709	17,605	168,31
1876	2,550 3,768	204,630	18,091	222,72
1877	: / : .			′
1878	3,012	154,757	16,909	171,66
1879	3,283	164,013	$\begin{array}{c} 24,637 \\ 22.941 \end{array}$	188,65
1880	5,495	278,022		300,96
1881	7,546	381,966	31.674	413,64
1002	7,521	363,835	25,536	389,37
1883	7,745	361,949	21,721	383,67
1884	7,204	286,393	1,863	288,25
1885	5,523	207,228		207,22
1886	4,168	164,111		164,11
_ 1887	8,442	331,621		331,62
December 31—		070.000		0.50 00
1888	9,200	353,990		353,99
1889	8,869	378.057		378,05
1890	12,798	594,746		594,74
1891	10,118	555,080		555,08
1892	11,677	667,775		667,77
1893	14,437	865,379		865,37
1894	5,814	225,720		225,72
1895	8,814	260,090		260,09
1896	15,230	437,159		437,15
1897	8,533	270,952		270,95
1898	13,482	743,820		743,82
1899	20,793	1,990,649		1,990,64
1900	14,417	1,390,141		1,390,14
1901	14,325	895,010		895,01
1902	18,201	1,168,554		1,168,55
1903	16,007	1,207,700		1,207,70
1904	12,674	905,581		905,58

# GERMANY

The graphite mines in the Passau district, Bavaria, produce the bulk of the graphite in that country, and in the following table the statistics of production and some figures for export are given:—

TABLE 29. PRODUCTION.

	Yəar.	Metric Tons.	Value.	
1893		3,140	\$52,010	
		3,133	45,732	
1895	· · · · · · · · · · · · · · · · · · ·	3,751	50,612	
1896		5,248	72,108	
		4.593	97,916	
1899		5,196	120,250	
		9,248	136,500	
		4,435	58,000	
		5,023	42,434	
		3,720	36,288	
		3.784	41.118	

<sup>\*</sup> From the British Embassy at Munich, Bavaria.

#### ITALY.

Most of the graphite mined comes from the Pinerola district of Piedmont along the Catian Alps. The production has been steadily increasing from 2,415 tons of a value of \$6,593 in 1892 to 10,313 tons of a value of \$55,660 in 1904. The bulk of the material produced is of the amorphous variety.

The following table shows the production of the mineral since 1891:—

TABLE 30. PRODUCTION.

· Year.	Metric Tons.	Value.
1891	2,415	\$6,593
1892	1,645	3,778
1893	1,465	3,080
1894	1,575	2,520
1895	2,657	8,599
1896	3,148	10,193
1897	5,650	11,300
898	6,435	17,423
.899	9,990	55,944
900	9,720	55,720
901		59,211
902	8,200†	46.740
903	7,920†	45.144
904	9,765†	55,660

<sup>†</sup> Mineral Industry and "Rassegna Mineraria" (Nov. 8, 1905).

<sup>‡</sup> Estimated value.

#### AUSTRIA.

The mines of the Bohemian forest and of Moravia produce most of the graphite, principally the amorphous kind.

The production and export of the mineral is shown in the following table:—

TABLE 31.

Year.	Production.		Export.	
	Metric Tons.	Value.	Metric Tons.	Value.
1891 1892 1893 1894 1895 1896 1897 1898 1899 1900	21,346 20,978 23,807 24,121 28,443 35,972 38,504 33,061 31,819 33,663 29,991	\$693,26? 637,012 637,870 881,980 985,771 1,216,458 541,058 349,426 395,280 418,126 363,702	11,985 11,536 8,812 11,923 13,091 14,229 17,109 19,451 18,996 14,900	\$258,168 251,166 176,948 200,104 219,676 228,698 252,238 283,771 296,648
1902 1903 1904	29,526* 29,589* 28,620*	363,169† 363,894† 352,026†	9,233* 10,076* 9,154*	238,600

<sup>\*</sup> Compiled from the report of the British Embassy at Vienna.

#### MEXICO.

TABLE 32.

Year.	Production.		Export.	
	Metric Tons.	Value.	Metric Tons.	Value.
1899	20,305		2,305	\$22,847 25,650
1900	2,561	\$25,600†	2,561	25,650
1901	1,473	15,000+	762	7,615
1902	580	5,800+	580	
1903	1,952	19.520†		

<sup>\*</sup> Compiled from the report of the British Embassy at Vienna.

## CEYLON.

According to figures given by Sir Le Neve Foster,\* in part four of the Mines and Quarries Report, the world's output of

<sup>†</sup> Estimated value,

<sup>†</sup> Estimated value.

<sup>\*</sup> Stonier, Graphite Mining in Ceylon, Inst. of Min., Eng., 1903.

graphite for 1901 was nearly 77,100 tons valued at nearly £785,000. Ceylon furnished 29% of this quantity and 80% of the value, and India contributed 3%. The figures of export and value for 1902 are as follows:—

25,189 tons of dressed ore valued at 10,516,366 rupees.\*

# Export Trade.

According to Mr. A. M. Ferguson,† graphite is mentioned in the Singhalese letters of the fourteenth century and in Dutch Government records of 1675. British records for 1831 give figures of export which must have commenced between 1820 and 1830, but it was not of importance until 1834 when it amounted to 129 tons valued at 12,054 rupees. In 1869 the output reached 11,306 tons valued at 889,620 rupees; in 1899, 31,761 tons valued at 22,255,400 rupees; and in 1902 it was 25,189 tons valued at 10,506,366 rupees. Details of the output for each year from 1834 to 1902 are given in the table below. Of the total amount, 96½% is exported from Colombo, and the remaining 3½% from Galle.

TABLE 33.
OUTPUT AND VALUE OF GRAPHITE IN CEYLON FROM 1834 TO 1902.

Years.	$\mathbf{Q}$ uant	ity.	Value.				
	Cwts.	Qrs.	lbs.	Rupees.	Cts.		
1834	2,582	2	14	12,054	00		
1835	4,952	3	0	11,082	50		
1836	12,644	0	5	14,663	50		
1837	3,700	0	0	4,293	00		
1838	1,164	1	12	1,379	00		
1839	423	1	18	490	00		
1840	981	0	0	1,225	00		
[841	2,002	2	7	2,684	50		
1842	7,285	0	3	12,317	00		
1843	3,677	3	20	5,238	50		
1844	9,914	3	21	12,946	00		
1845	19,245	0	15	24,519	50		
1846	25,036	3	7	30,361	00		
847	9,248	3	11	10,583	50		
848	6,787	0	0	7,062	00		
849	3,329	2	20	3,302	00		
850	23,021	1	6	38,330	00		
851	23,865	1	2	52,554	00		
852	13,110	1	21	26,281	00		
853	19,577	2	25	40.572	00		
854	17,451	2	19	39,162	00		
855	6,129	$\bar{3}$	16	11,448	50		
856	13,380	$\tilde{2}$	27	33,380	00		
857	33,497	ō	4	83,850	00		
858	19,432	3	12	33,841	50		

<sup>\*</sup> One rupee=1s, 4d.=34.6 cents.

<sup>†</sup> Royal Asiatic Society, 1900, Vol. IX, part 2.

TABLE 33.—Continued.
OUTPUT AND VALUE OF GRAPHITE IN CEYLON FROM 1834 TO 1902.

Years.	Quant	ity.		Value.				
	Cwts.	Qrs.	lbs.	Rupees.	Cts.			
.859	17,510	3	11	41,138	00			
860	75,660	ŏ	23	239,535	50			
861	38,345	ĭ	23	110,643	50			
862	40,895	3	13	130,789	50			
863	65,128	ő	3	281.246	00			
	84,028		4					
864		2	5	404,314	50			
865	40,143	3		151,206	00			
866	56,278	3	14	218,605	50			
867.,	45,836	0	14	193,601	00			
868	141,095	0	14	720,410	50			
869	226,131	3	8	889,620	00			
870	85,248	3	18	345,622	00			
371	125,257	1	5	620,953	50			
372	136,051	$^2$	23	438,366	64			
873	173,996	0	17	1,479,395	44			
874	149,938	1	3	1,440,166	87			
875	110,023	1	0	1,100,232	53			
876	117,361	1	2	1,173,612	64			
877	96,792	1	21	967,924	37			
878	84,634	3	15	846,348	84			
879	162,495	$\tilde{2}$	$\overline{24}$	1,624,957	15			
880	205,738	$\frac{2}{2}$	9	2,057,385	81			
881	259,909	ō	16	2,599,091	42			
882	260,166	1	6	2,601,663	3			
	262,773	3	1 :	2,627,737	58			
883	182,425	3	10	1,824,258	40			
884	196,399	2	23	1,963,997	06			
885	241 760	õ			15			
886	241,760		13	2,417,601				
887	238,599	3	0 1	2,385,997	50			
888	223,277	3	4	2,232,777	82			
889	486,138	3	1	4,861,387	59			
890	392,577	2	13	3,925,776	16			
891	400,540	0	15	4,005,401	34			
892	430,666	3	20	4,306,669	28			
893	332,168	3	$16\frac{1}{2}$	2,491,266	72			
894	335,168	0	24	2,513,761	60			
895	326,754	1	16	2,450,657	93			
896	361,061	1	13	3,069.021	62			
897	379,415	$^{2}$	21	3,670,846	- 78			
898	478,318	0	2	7,174,770	27			
899	635,224	1	5	22,255,400	77			
900	391,699	3	6	9,792,495	09			
901	446,960	_		9,609,642	_			
902	503,778	_	_ 1	10,516,366	_			
	*482,105	_		10,750,941	_			
903	*521,204	_		11,622,849				
					0.0			
Total	11,360,029	2	23	147,760,072	90			

Note.—The figures for the years 1834 to 1884 are taken from the Journal of the Royal Asiatic Society, 1887, vol. ix., part ii.; and for the remaining years from the blue books of the Ceylon Government. From 1836 to 1845, and from 1858 to 1868 inclusive, the customs duty was 2½ per cent. ad valorem; from 1846 to 1857, inclusive, there was no customs duty, by No. 9 ordinance of 1847; and from 1873 to 1883, inclusive, licenses to dig for plumbago cost 10 rupees each, with a royalty of 10 per cent. of the value of the plumbago dug, in the case of the western province.

<sup>\*</sup> From the Royal Colonial Institute, London.

The foreign countries to which graphite was exported during the years 1885 and 1902 are shown in the table below; the large increase in the amounts sent to the United States, Germany and Belgium will be noted.

TABLE 34.

EXPORTS OF GRAPHITE IN YEARS 1885 AND 1902.

Destination.	1885	1902
	Cwts.	Cwts.
Great Britain	136,964	135,47
United States.	54,891	272,21
Germany	1,199	68,44
Belgium	400	19,56
rance	1,237	1,8
Australia	1,176	1,49
Iolland		1,1
apan		1,10
Russia		1,0
ndia	306	7
weden		4
urkey in Asia.		19
taly		-
New Zealand		
traits Settlements		
Austria	226	
	196,399	503,7

## INDIA.

The production of graphite in India since the year 1895 has been as follows\*:—

TABLE 35.

Year.	Tons.	Year.	Tons.
1895 1896 1897 1898	1,597 306 93 194 332	1900	1,829 2,727 4,882 3,648

<sup>\*</sup> From the Royal Colonial Institute, London.

# THE WORLD'S PRODUCTION OF GRAPHITE.

The world's production of graphite for the year 1903 will be seen from the following table:—

TABLE 36.

Country.	Metric Tons.	Dollars.
Austria	29,590	363,894.00
Ceylon	24,105	+3,583,646.00
Italy.	7,920	45,144.00
Bavaria.	3,720	32,288.00
India	3,648	36,000.00
United States.	2,053	225,554.00
Mexico	1,952	19,520.00
Canada	728	23,745.00
Japan	114	11,400.00
Sweden	25	2,500.00
_	73,855	4,343,691.00

# CONSUMPTION OF GRAPHITE BY THE VARIOUS BRANCHES OF $\frac{\text{MANUFACTURE.}}{\text{MANUFACTURE.}}$

In the following table the estimated proportionate amounts of graphite used for different purposes are given:—

TABLE 37.

	Manufactures		Per Cent
Pofractory articles as or	ucibles, stoppers, nozzles, etc.		35
Remaciory articles as cr			30
ubricating graphite		!	10
Joundry facings			8
Ironhite grange			6
Popul load			4
Teachite peeling			2
rapine packing	llaneous	1	3
rapinte paint	11		1
Slectrotyping and misc	lianeous		1
	•		

About 55 per cent. of the crystalline graphite is used for the manufacture of crucibles, retorts, and other refractory apparatus; in Europe several large steel works use to some extent the amor-

phous variety for this purpose. About 15% of the total production is absorbed by stove polish; about 10% by foundry facings, and 5% by graphite paints (the bulk of the graphite used for this purpose being of the amorphous variety), and about 15% by the manufacture of pencils, electrotyping, steel packing, and electrical supplies. In the United States and Canada, with the exception of the Georgia amorphous graphite, which is used in coloring fertilizers, the bulk of the amorphous graphite mined is employed in the manufacture of paint and for foundry facings.

## PRICES.

On account of the great variety of grades produced in the graphite mines and mills, it is next to impossible to generalize on the subject of prices. In every country the requirements of the trade demand specific articles and a comparison of the various qualities for the purpose of arriving at average values would be a task of great difficulty. These prices could be of no service to those requiring to purchase or needing for service a specially prepared graphite, and in this case the consumer will have to apply direct to the graphite producer. However, an endeavour has been made to give, in the following, a list of average prices generally paid for grades mostly in demand:—

#### CANADA AND UNITED STATES.

Graphite for refractory articles as crucibles, stoppers, nozzles, etc., f.o.b. works: from 5 to 9 cents per pound.

Graphite for foundry facings, from  $1\frac{1}{2}$  to 4 cents per pound.

Graphite for paints, from 1 to  $2\frac{1}{2}$  cents per pound.

Graphite for lubricating, from 6 to 10 cents per pound.

Graphite for pencils, from 4 cents per pound.

If lubricating stocks are put up in individual packages of small weight, as high as 16 cents per pound may be obtained.

## BAVARIA AND BOHEMIA.

The following prices have been obtained from dealers in Nuremberg and Vienna for both the Passau and the Bohemian qualities: Graphite for the manufacture of crucibles, containing 91° carbon, 64.10 marks\* per 100 kilos.

Graphite for the manufacture of pencils, with 83% carbon, much used by pencil manufacturers in Nuremberg, 15.75 marks per 100 kilos.

Graphite, used principally for stove polish, with 72% carbon, 10.20 marks per 100 kilos.

Graphite used for stove polish and paints, with 52% carbon, 9.40 marks per 100 kilos.

#### CEYLON.

For commercial purposes the graphite is graded,

- 1-large lumps.
- 2-ordinary lumps.
- 3—chips.
- 4-dust.
- 5-flying dust.

The ranges of size and price may be gathered from the following recent quotations:—

														Per Ton.
arga lumpa	 							 			 	 	 _	\$ 100
Large lumps Ordinary lumps	 	 	 •				 						 	65-190
Ordinary lumps hips Oust	 												 	50-125
Just	 											 	 	15 70

Regarding the Ceylon qualities there is a call for more uniform standardization. The finest plumbago, soft, lustrous and greasy, often found in flaky and circular form, is termed "X" and "X,B." Lower grades, owing to the hard, brittle, dull and stony forms, are indicated by various letterings. The poorest of all, often containing 25 per cent. of clayey matter, is called "bora." Dust and flying dust contain a considerable percentage of sand and other impurities. The variation in the qualities of the mineral

<sup>\*</sup> One Mark approx. 25 cents.

<sup>†</sup> Ralph Stokes, Mineral Resources of Ceylon, Mining World, 14th April, 1906, p. 464.

is based on physical differences, not chemical. Even in two parcels of plumbago, realizing £20 and £40 per ton respectively, the percentage of carbon may be the same. Thus the differentiation is not based on conditions determinable with scientific precision, but is largely dependent upon personal judgment. Only years of practical experience can make a trustworthy and rapid judge of qualities, whose duties are often made the more difficult by the mixture of grades, the poor appearing bright and lustrous after its natural "black leading" by the good. Buyers still have occasion to complain of appreciable disparity between sample and bulk consignment. This may be due to the difficulty of maintaining standards of scientific equality, and also to the business caution of the Singhalese merchants, who must unfailingly receive the benefit of every doubt.

## CHAPTER VIII.

# DRESSING AND REFINING OF GRAPHITE.

The now exhausted mines of Cumberland produced a graphite of such purity and solidity, that it was only necessary to cut the same in small strips for the manufacture of pencils or apply it to other purposes in its natural state. It is doubtful whether graphite of such purity is found at present in any other locality. As a general rule the graphite ores are not in such a rich state, when they come from the mine, as to admit of their being at once sent direct to the market; they contain more or less impurities such as oxide of iron, silica, alumina and lime or are so hard that they cannot be employed at all in their natural state. They must be enriched by some means or another so that the worthless gangue or country rock may be got rid of, thus increasing the quantity of carbon in the ore. The methods of enriching or refining graphite may be divided into three general systems.

1. Hand sorting. 2. Mechanical separation. 3. Chemical refining. The mechanical separation is again subdivided into the dry or wet method.

As to hand sorting, this is practised in a very efficient manner in the Austrian and Bavarian mines, while no or scant attention is paid to this particular branch of ore dressing on the North American continent. The choice of one or the other systems in the mechanical separation or chemical refining depends entirely upon the nature of the ore to be treated and the purposes to which the finished product is to be applied, though too often the selection is based on chance, prejudice and limited experience. The selection of the most profitable process and machinery for the treatment of a given graphite ore is of great importance in a mining enterprise; but as some ores are susceptible of successful working by more than one process—and in such cases local conditions must determine which methods will yield the best results—it is not possible to lay down exact rules covering all cases.

# HAND SORTING.

In most of the mines it is of great service to subject the mineral to a hand sorting process before it is delivered to the mill. It saves

the expense of dressing and in some cases also of shipping waste rock, and at the same time increases the capacity of the mill. It is specially useful in the case of pure vein graphite, which is sent very often to the market without being further subjected to mechanical treatment. Of great importance, also, is its application to disseminated graphite ores; because sometimes lean portions are mixed with the useful ore in mining, and, if not separated, will heavily tax the mill. The picking out of wood, rope ends, bolts or pieces of tool iron is also very important in order to rid all the apparatus of those troublesome stoppages, that cause so much derangement of mill work and annoy the millmen.

Separation of the gangue from the ore is practised to some extent as a part of the mining operations, as in the Bavarian mines where the miners are trained to cull certain portions of rich ore, carrying over 60% of graphite, from the accompanying gangue, while all the waste work is picked out and left in the mine for filling up the stopes. Local conditions, of course, dictate in most cases to what extent the cobbing of the ore shall be continued, before it is delivered to the mill.

The process of hand sorting is divided into two operations. 1st. Breaking of the ore and 2nd. Handpicking. The former is done by a jaw-crusher or manually with the aid of a hammer. As a rule the breaking of an ore by a jaw-crusher produces an excessive quantity of fines, which is not desirable for hand sorting, and generally the breaking with the aid of a hammer is much preferred. The size, to which the ore has to be broken in order to make hand sorting very efficient, depends upon the general character of the same and no rules can be laid down.

After the ore is broken either by machine or with the aid of a hammer it is passed over a screen or a grizzly to remove the fines, which are sent immediately to the mill for future treatment. The remaining pieces of ore are sorted either by men or boys. In Europe boys are frequently employed for such work; they become very expert in this work and are satisfied with much lower wages than men, while they are even more alert and efficient. The success of hand sorting is largely dependent upon the manner in which the ore is presented to the sorters. For this purpose the latter may sit along large stationary tables, on which they may draw from heaps or pockets the ore to be sorted; an efficient method is by discharging the ore on a circular table revolving slowly, around the periphery of which the sorters stand; the ore

may also be discharged on a travelling belt, from which the sorters can select it.

A revolving picking, table, as manufactured by the Allis Chalmers Co. of Chicago, is illustrated in Figure 17. The broken

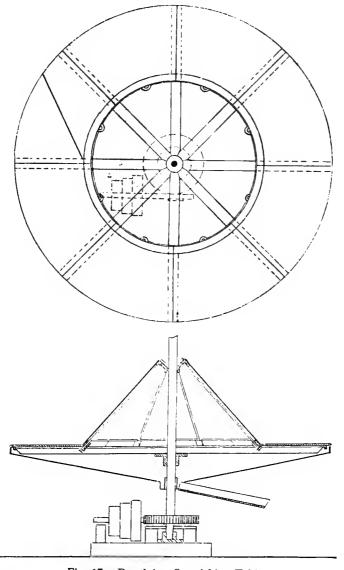


Fig. 17.—Revolving Ore-picking Table

ore is placed on the table by a chute, and the spreading out of the same is carried out by the revolution of the table until meeting an incline stationary scraper; it is swept off into a chute which delivers it into cars or a continuous conveyor to transport it to the next operation. Boys are stationed around the table, who pick out the mineral from the slowly moving layer of ore. The table is made of punched iron or steel plate. Experience has shown that it is easiest for the pickers to throw the sorted material in front of them and this arrangement is easily made with tables of annular form in connection with which a conical surface may be arranged inside the ring, around the vertical axis, with radial partitions in order to facilitate the separation of different classes of ore, which will slide down the cone into proper receptacles.

The endless belt tables are so well known that they require no special description beyond the statement that they are often inserted in the mills between the crushing machinery. The Robins Belt Conveying Company makes a special picking belt, which is used in a number of mills; it is heavy and from 32 to 36 inches in width, and is supported on idlers which are so shaped as to give the belt a broad flat surface at the centre, with slightly raised sides. It is made to travel at speeds varying from 30 to 60 feet per minute. Owing to its elasticity the belt will withstand spalling of the ore directly upon its surface. The advantage of rubber belts, over any other make, lies in the fact that there are no links to wear and no crevices wherein pieces of ore can jam.

In order to secure maximum efficiency in hand sorting, it is necessary first to have good light, since in many cases the richer portions of graphite ore are distinguishable from the leaner portions with difficulty; further, good supervision and all arrangements which tend to increase the convenience of the pickers. One point is very essential, and that is that the layer of ore must be put on the table in such a way that every piece can be noticed and that every part of the material is within reach of the sorters.

In Europe, especially in the older works of the Bohemian forest, culling is carried on to a degree of subdivision, which would not be done in modern practice. Under ordinary conditions culling would be a step in the milling process, all the ore from the mine, having been broken by a crusher to the size determined for the next machine, would pass over a grizzly from which the coarse

material would go to the picking table and the rejected stuff from the latter to the next crushing machinery.

#### MECHANICAL SEPARATION.

Before entering into a description and general outline of the various methods in use, it is necessary, in order to fully understand the working principles of the same, to describe the different classes of apparatus which, according to experience, have given satisfactory results in the mechanical separation of graphite. It must be mentioned, however, that the list of the machinery enumerated in this chapter is not by any means an exhaustive one, since in the majority of mills the construction of special apparatus for the manufacture of certain grades, for private reasons is kept secret, and information regarding the same is not available. With the exception of some of these apparatus of special design, the machinery usually employed in the mills does not differ materially from those employed in the mechanical separation of other ores, but its operation and adjustment require in a few cases special attention.

# MACHINERY USED IN THE DRY METHOD.

# Drying of the Orc.

It is seldom that an ore freshly mined is dry enough for immediate treatment by the dry process; most of the run of mine carries a good deal of moisture sometimes as high as 15%. There are three ways of drying ore;

- 1—By exposure to the air.
- 2—By means of steam pipes.
- 3-By direct heat.
- 1.—Drying by exposure to the air:—The material is spread over a large wooden platform in a layer two or three inches in thickness. If the weather is favourable a sufficient amount of moisture evaporates, naturally, to render the mineral fit for treatment by the different processes of crushing and blowing, but a wet season interferes with the work, while drying by this process during the winter is impossible especially in severe climates. This method is unsuitable and unreliable and for this reason its application is very limited.

2nd.—Drying by steam pipes:—A number of 1½ and 2-inch steam pipes are arranged parallel to each other and close together on the floor of a shed, joined at the ends to form a continuous length, one end terminating in a pipe of large dimension connected with the exhaust of some steam engine, the other end leading into the open for the discharge of the steam. Where much material is to be handled, a space is generally left in the middle of the shed for a track which allows the ore to be unloaded at any point desired. All dried material is shovelled into an elevator placed at a convenient point near the track, which delivers the same to the crushers of the mill. This arrangement has proven satisfactory in some mines where the ore does not carry much moisture; no extra power nor fuel is required, while there are hardly any repairs and the danger from fire is completely eliminated.

3rd.—Drying by direct heat:—By this is generally meant the use of a rotary drier or of a natural draft gravity flow drier. method by direct heat is by far the quickest for a large tonnage. for the reason that a temperature of 2.500 to 3.000 F. degrees can be obtained, while by using direct steam heat the temperature is only 230° to 330° F. degrees, and this temperature is in the inside of the pipes, which means a considerable lower temperature on the outside where the material lies. Great care, however, should be taken in the construction and direction of all direct heat dryers, otherwise no end of trouble will arise; all iron parts should be so constructed as to allow for expansion and contraction, otherwise they will soon break. All settings and bearings for the dryer should be extremely substantial on account of the liability to get out of place by the settling of the brick work and by the extreme heat. Great care must also be taken with every joint or rivet, otherwise the contraction and expansion will soon cause leakage. In drying, all ore should first be broken into two inch cubes or less, as it does not pay to dry large pieces; so let it be understood that all material is supposed to have been crushed before entering the dryer.

## ROTARY DRYER.

The rotary dryer, as illustrated in Plate XIV, consists of a long cylinder made of strong boiler plate, resting and turning on its ends on friction rollers; in order to allow the shell to expand, and at the same time to prevent it from sliding, these friction rollers are flat

# PLATE XIV.



Rotary Dryer.

at the upper end and grooved at the lower end of the cylinder. The length of the shell is from thirty to forty feet, the diameter from two and a half to four feet, and its inclination 7°. The whole is bricked in, leaving only the ends of the cylinder with the friction rollers outside. The space between the arch and the cylinder is six inches.

The drying is assisted by longitudinal blades, which lift the material and allow the same to fall through the current of hot air which circulates through the cylinder. The fire is either placed directly under the shell, or in an extra brick case at the side or on the lower end of the cylinder, allowing the heated fumes to play round the shell and escape through a chimney placed at the other end of the dryer.

Sometimes fires are made at both ends, the chimney in this case being placed in the middle of the apparatus. The cylinder is made to revolve from six to eight revolutions per minute. The ore, which is charged by hand or by automatic arrangement, travels along very gradually, is stirred up by the inside blades, and, as a rule, discharges into the elevator for the ore bin. The capacity of this rotary dryer ranges from fifty to seventy-five tons per shift, according to size and the contents of moisture in the material. The cylinder is kept in motion either by an endless chain round the lower end or by gearing.

The main advantage of a rotary dryer over all other drying methods is its continuous operation and the handling of a large quantity of ore in a comparatively short time.

Where the charging is done automatically, one man is sufficient to attend to the whole apparatus, otherwise two men are needed. The expense for fuel per shift for a dryer of the dimensions represented in Plate XIV, is about \$3.50, on a basis of \$2.00 per cord of soft wood, and \$3.00 per cord of hard wood.

# NATURAL DRAFT GRAVITY FLOW DRYER.

This dryer, Fig. 18, consists of cast iron plates bolted together to form a steep inclined chamber having a feed hopper at the top and a discharge gate at the bottom. The under side of the chamber consists of overlapping steps which extend from the hopper to the outlet. A large air space is provided between the lower edge of each step and the upper edge of the next, through which space the heat from the fire pot flows upwardly through the rock

as the latter flows down the steps. A dam opposite each step prevents the rock from running down faster than the speed at which the discharge is adjusted by setting the outlet gate. This dam is adjustable to determine the depth of rock on the steps, and is high enough above each step to permit free flow and insure against clogging.

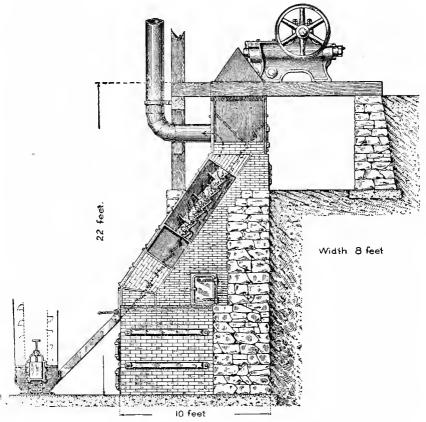


Fig. 18.—Natural Draft Gravity Flow Dryer as manufactured by the Krom Machine Works, Jersey City, N. J.

The rock is fed from a rock breaker or otherwise into the hopper, and runs down the steps by gravity in opposition to the upflowing heat, which is raised from the fire pot and drawn up through the rock at every step by the natural draft of the stack. Thus natural forces are used to move the rock and the heated air against each other in opposite directions, avoiding all moving or driven parts.

The dryer is so compact that it can be set on any floor space 8 x 10 ft. and within a height of 18 ft. The furnace can be bricked in with the doors at either side or at the back. This makes it possible to use the dryer where space is limited.

#### ROCK BREAKERS.

The rock breakers employed are of two classes:

- 1. The jaw breakers, which are intermittent machines.
- 2. The rotary and the spindle or gyrating breakers, which are continuous machines.

## Jaw Breakers.

The first crusher through which the rock has to pass is invariably a jaw crusher of large size. This is a machine for reducing rock preparatory to fine crushing by rolls. It is durable and simple to operate. The rock is crushed between jaws, one stationary, the other swinging and driven by a powerful toggle movement.

The adjustment of the jaws and the size of the rock leaving the crusher is determined by the character of the apparatus used in subsequent treatment. One rock crusher alone may be used to prepare the rock for the rolls, or other fine crushing machinery, but for a larger capacity it is preferable to use two sizes with a screen between, the second crusher relieving the subsequent apparatus of a great deal of work.

Since the large size and the irregularity of the feed rock generally does not admit of automatic feeding, the jaw breakers are fed by hand and shovel; in many cases by a shute, sloping from the bottom of a bin, the attendant pulling forward the ore in the shute by a rake or pick.

The jaw breakers may be divided into two different types, according to the movement of the jaws:

1st. Those which are pivotted above giving the lower part of the jaw the greatest movement.

2nd. Those which are pivotted below giving the upper part of the jaw the greatest movement.

To the former class belong the Blake crushers, to the latter the Dodge crusher.  $\bullet$ 

The movement of the lower part of the jaw is greater in the Blake crusher, and the result is that a product of various sizes must

drop from the machine, whereas in the Dodge crusher the movement is greater at the top of the jaw, the lower part remaining nearly stationary, and the product leaving the machine must be of nearly uniform size, determined by the distance the jaws are set apart. This explains the higher capacity of the Blake, while the Dodge crusher delivers more fines and a more uniform product.

Of the two kinds of rock breakers just described it appears that the Dodge crusher is generally preferred in graphite mills, since this apparatus delivers (all other conditions being equal) more fines.

# Rotary and Gyrating Crushers.

These crushers are also used in graphite mills, and as their working principle is so well known or can be easily studied in the catalogues of the manufacturers of this class of machinery, a description of them is omitted. It might be said, however, that the application of these crushers to graphite ores, especially to such ores which consist of pure or nearly pure graphite, or which contain a slippery gangue, is restricted, owing to the loss of efficiency caused by the slippery condition of the material.

## FINE CRUSHING AND PULVERIZING.

The machines for final crushing receive the ore from the rock breakers, and are suitable for liberating the graphite from the waste preparatory to concentration. They act on the principle of crushing by direct pressure, as in the rolls or by centrifugal force as in the ball mills.

## ROLLS.

The chief parts which enter into the construction of a pair of rolls, are a pair of shafts upon which are usually mounted permanent cores of soft cast iron, carrying shells of rolled steel or chilled iron, which constitute the crushing surfaces; one shaft revolves in fixed, the other in moveable boxes.

In some rolls the shells are made of manganese steel, which has an extraordinary hardness and toughness, and which lasts much longer than those made of ordinary or chilled iron.

The shaft in the moveable boxes is held towards the fixed boxes by powerful springs, the degree of approach being regulated generally by compression bolts. The space between the rolls varies from practically nothing up to three-quarters of an inch. The relation between the diameter of the ore fed and the space between the rolls, that is to say, the amount of reduction, is most important, if the rolls are to do their work. In some mills the ore is charged in nut size, in others in much smaller lumps and the spaces between the rolls are adjusted accordingly.

They are however fed rarely with lumps larger than one and three-quarters of an inch in diameter, generally with an admixture of fine grains. In taking the rock for the rolls from a breaker or other apparatus, the supply of ore is regulated and the output is limited, but it often happens that a sudden rush of ore will choke the rolls, and unless they are supplied with an extraordinary amount of power and strength, which is generally not the case, they will break, especially those which are driven by gearing motion and pulleys.

To avoid this trouble, in some mills, feeders are used kept constantly full of material, which is fed to the rolls by an oscillating gate. Small scrapers are also used in several cases to remove the adhering fines from the face of the rolls at the lowest point in their revolution.

The design in crushing rolls in which one is made two or three inches wider than the other, so that the narrower may wear into the wider, forming a flange on either side is bad. Equally bad is the design in which one roll is purposely made with a flange. These designs increase the friction, decrease the capacity of the rolls and increase the cost of maintenance.

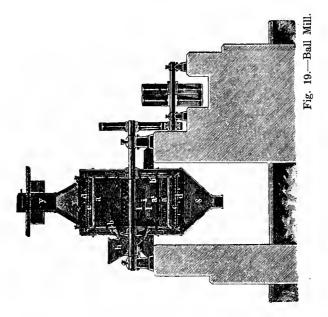
Graphite ores being as a rule very slippery, having a small coefficient of friction, it is necessary that the space between the rolls be set wider, or the lumps fed must be smaller than those of ordinary ores.

For general purposes crushing rolls are made in five sizes from 12 by 10-inch to 36 by 18-inch rolls. The minimum and maximum capacities are one and eight tons per hour, according to the desired product, and the horse power required from five to twenty; all rolls make from 125 to 150 revolutions a minute.

### BALL MILLS.

There are a number of designs on the market, for which great effect and capacity is claimed in pulverizing, but the writer will confine himself to a description of only two machines which have been successfully used in graphite mills; the Krupps or Gruson ball-mill and the American ball-mill.





The Krupps or Gruson ball-mill is shown in Figure 19. It is a cylindrical mill revolving on a horizontal axis. In the interior of the cylinder are a large number of chilled iron or steel balls of different sizes. The principle of pulverization is based upon the attrition of the balls against each other and against the die ring. The latter is composed of fine perforated spiral chilled iron plates arranged so that each laps the next, which by forming steps give

the balls a drop from one to the next and furnish a space beneath the steps for the return of the oversize of the outer screens. Outside the die plate is a coarse perforated screen in five parts with spaces between to take the wear and again, outside that there is a fine gauze screen. At the end of each section of fine screen a deflector or shovel is placed to convey in its upward journey the oversize of the screens back into the grinding space. The ore is fed through a hopper at one end and is discharged through the screens. The cylinder is enclosed in a plate iron housing with a discharge spout below. The mill is driven by gear and pinion with tight and loose pulleys; it can be run dry and wet and is specially adapted for graphite ores. The ore is charged in egg size.

In dry crushing, if it is desired to remove the fine graphite dust, the upper funnel is connected with an exhaust fan which carries away the light impalpable powder, to be deposited in a dust chamber. All the working parts are made of the best chilled iron and cast steel, and the makers claim that, owing to their large experience in steel making for armour plates and war material, they have succeeded in turning out a metal which will resist the wear and tear inseparable from a pulverising machine.

The American Ball Mill:—This mill is illustrated in Fig. 20 and 20a, and consists of a cast iron casing A attached to the vertical axis B, driven by pulley G and revolves in the manner indicated. Inside the casing is an iron plate C having at its periphery (see Fig. 20a) four semi round holes for the reception of four steel balls. each weighing 6 kilos. This plate revolves in an opposite direction to the iron casing and is driven by pulley D. The mill proper is surrounded by a casing E, made of galvanized iron and having a feeder H on top near the centre and a discharge J below the mill. The wear and tear of the balls is prevented by heavy steel pins K, placed in the manner indicated in the iron plate C. The working of the mill is as follows: The graphite ore, charged in the centre through feeder H, is thrown by the centrifugal force of plate C towards the die ring of the mill and is crushed and pulverized by the force of the heavy steel balls moving in opposite direction. The finely pulverized material is discharged towards the centre through the openings L, while the coarse stuff is continually exposed to the crushing force of the balls. This mill is specially adapted for pulverizing graphite and has given good results wherever employed.

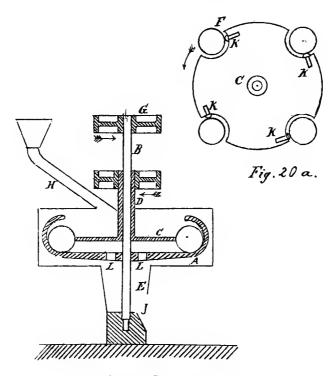


Fig. 20.—Cannon Ball Mill.

### EMERY MILL.

Another pulverizer which has been used with satisfaction for grinding graphite ores is the so called Emery-mill.

It is well known that the skirt or outer margin of buhrstones, described on page 174, wear faster than the eye. These stones correct that difficulty by having an eye of buhrstone and a skirt of emery. This emery millstone is prepared with an iron cup or shell, into which are placed blocks of emery which are laid as a skirt around an eye of buhrstone, and slabs of sandstone on edge are placed in positions through the emery skirt corresponding to the furrows of the buhrstone; then the melted (zinc, bronze, or iron) is poured around the blocks of emery and sandstone, fastening them firmly in their places. These millstones need but little dressing, the furrows are easily opened, because they are made of softer material than the rest.

These mills, as illustrated in Fig. 21, are made either with horizontal or with vertical stones. In the horizontal Emery mill the bedstone A is bolted strongly to the top case B and is lowered with it directly upon the runner stone C, with which it is then in perfect adjustment. The clamp-ring D is then tightened

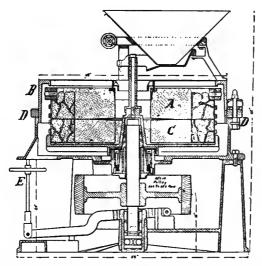


Fig. 21.—Horizontal 42 inch Direct Running Emery Mill as manufactured by the Sturtevant Co., Boston, Mass.

and grasps the bedstone case, firmly holding it and its stone immovably in position. The runner can now be lowered away from the bedstone by the hand wheel E to such a distance as gives the fineness of grinding required. This simple and accurate adjustment of the millstones is of special value, since it insures good results with ordinary help. The stone makes from 300 to 350 revolutions per minute; the capacity of a 42-inch mill is from one to three tons per hour and the approximate horse power required is eighteen.

In the vertical Emery-mill the adjustment of the stones for coarse or fine grinding is accomplished by turning a hand wheel at the end of the shaft. A 30-inch mill having a capacity of from two to four tons per hour, according to fineness desired, requires from eighteen to twenty horse power to run it and makes about 650 revolutions per minute.

### BUHRSTONE MILLS.

In many graphite plants Buhrstone mills are used for the polishing and also grinding of flakes. Fig. 22 represents a Buhrstone mill which has given good satisfaction on graphite ores.

A represents a cast-iron frame, on the upper part of which is a cylindrical shell, B, to receive the runner or under stone, R. This shell is of larger dimensions than the stone, R, so as to leave a space all around and underneath the stone, R, as shown clearly in Fig. 1. The shell, B, has its upper edge made perfectly smooth and even, so that all parts of its surface will be in the same plane.

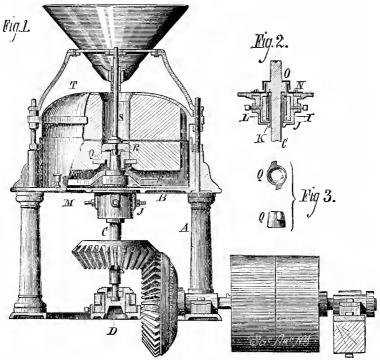


Fig. 22-Buhrstone Mill, manufactured by Muson Brothers Co., Utica, U.S.A.

In the lower part of the frame, A, there is placed a horizontal driving shaft, which has a bevel wheel secured to its end. This wheel gears into a bevel pinion, on a spindle, the lower end of which is stepped in a socket, D, the upper end of which has a flange around it. This socket is fitted with an adjustable box,

which rests upon a lever supported by a nut and screw, by which means the stones may be made to run at a greater or less distance from each other to vary the fineness of the flour.

The spindle, C, is provided with a collar, L, Fig. 2, which is fitted within a box, J, attached to the under side of the shell, B. This box, J, is of cylindrical form, concentric with the shell, and within it there are placed bearings which are adjusted snugly against the collar, L, by keys, screws, or other means. The collar is hollow and opened at its lower end, having a space all around between it and the spindle. The box, J, is provided with a central vertical tube, K, around which the collar, L, works, the tube, K, passing up between the collar, L, and the body of the spindle, as shown clearly in Fig. 2.

The driver is fitted on the upper part of the spindle, C, and like the clearer, is secured to the spindle by a feather and groove. The driver rests on the eye, Q, and has two arms projecting from its opposite sides, as shown in Fig. 3. The arms are rounded at their face sides or bearing surfaces, the curvature being in a vertical plane. The arms of the driver fit within recesses of a shell, which is secured concentrically within the runner, and has a pendant bearing which rests upon the apex of the spindle. The dansel, S, is attached to the upper surface of the eye, as shown in Fig. 1.

T is a cast metal cylindrical cap, in which the upper stone is secured by set screws. The cap is turned true at its lower part, so that it may fit into the shell, B, the cap being provided with a shoulder or flange all around it, which flange is parallel with the lower edge of the cap. The stone, Q, has an eye made in it, centrally, and the cap is secured in proper position by means of screw rods and nuts, the rods being attached at the shell, B, and passing through eyes at the outer side of cap T. On the cap, T, a hopper frame is placed, containing the hopper and shoe, which may be arranged as usual.

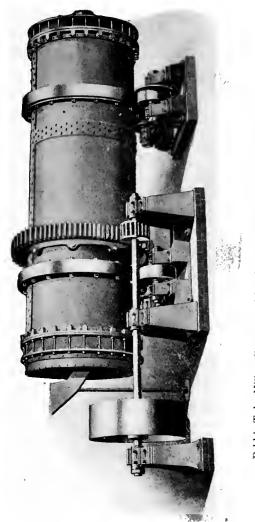
It will be seen from the above description that the runner will, in consequence of the arrangement of the driver, relatively with the apex of the spindle, be allowed to adjust itself to the stone, so that the parallelism of the faces of the two stones may be preserved as the stone rotates. This arrangement, to wit: the having of the apex of the spindle in line with the bearing surfaces of the arms of the driver, admits of a universal joint movement of the stone, an effect which cannot be attained in the ordinary arrangement.

## PEBBLE TUBE MILL.

This mill on account of its great efficiency and smooth action has found its way into graphite mills of recent date; it is used for polishing and grinding graphite flakes and can be so adjusted that the latter are freed from sand; it replaces effectually the old Buhrstone mill, while other mills either crush, twist or cut the material. Tube mills grind principally by friction, the effect being produced by the sliding, tumbling and rolling inside of the mill of a great number of flint pebbles or porcelain balls, which are mixed with the substance to be ground and the movement being caused by revolving the mill at a regular speed. This method of reducing materials has not only the advantage of being simple and power saving, but greatly economizes in labor, needing attention only while charging and discharging by unskilled work-No parts require dressing or sharpening as in all other grinding mills, as practically there is scarcely any wear, consequently the mills are always in good working order. These tube mills are not crushers, consequently all materials should be crushed to a certain fineness before being charged into the machine, in order to secure the best results. One great advantage in these tube pebble mills lies in the arrangement that the ore to be ground is fed in at one end and delivered as a finished product at the other end, the fineness of the product being regulated simply by the speed at which the ore is fed into the machine. It is claimed that a given quantity of product from a Tube mill contains at least 50 per cent. more extremely fine particles than a similar quantity reduced in any other existing form of grinding machine. As every particle of material must pass under the grinding action of the entire charge of pebbles a thorough and uniform grinding is bound to be the result; it is claimed that the uniformity of the product is so great that it is unnecessary to make use of sieves.

An illustration of this Pebble Tube mill, as manufactured by the Abbe Engineering Company, 220 Broadway, New York, is given in Plate XV. It consists of a long cylinder made of strong boiler plate, from 20 to 25 feet long and with a diameter ranging from 4 to 6 feet. The entire cylinder or tube rests on friction rollers and is kept in motion in the manner indicated in the cut.

The speed at which the mill revolves tends to carry the mixture of pebbles and ore to a certain height within the mill, from which point the pebbles and ore fall together, rolling and grinding as they seek the bottom of the mill.



Pebble Tube Mill as Constructed by the Abbe Engineering Co., of New York.



Discharge and Feed of the Abbe Tube Mill.

The mill is fitted out with spiral feed and discharge, shown on Plate XV. This arrangement does away with a great many troubles experienced heretofore; it requires no stuffing boxes and no manholes in the shell (which are always an obstruction to making a perfect lining inside of the machine.) The illustration shows both the discharge and feed end of the tube mill open. Over the outside surface of the spiral there is a one inch thick plate which is held in position by the bolts shown without nuts in cut. This plate is perfectly flat, having an opening near its circumference, the opening ending where the spiral starts: Through this opening the material passes into the spiral and is fed to the mill. In front of this plate is fastened a wrought iron receiving chamber, into which the material can be fed by a chute from a bin or other means convenient to the user.

### CONCENTRATORS.

In the dry concentration of graphite a number of appliances have been invented, but as information regarding the working principles and construction of most of them is not available, owing to the secrecy of the owners, the writer is able to furnish only a description of a few of them, which have been put on the market and which appear to have been applied with some measure of success. These appliances may be divided into air separators, which separate the particles by air blast and those which project the particles by a force other than air blast. To the former class belong the air jigs and to the second all the centrifugal concentrators.

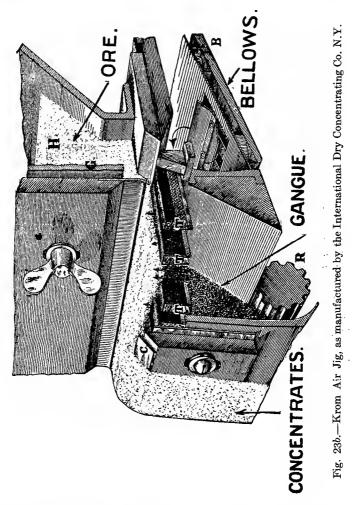
#### SEPARATION BY AIR BLAST.

# Air Jigs.

Krom Pneumatic Jig:—This apparatus is employed in a number of modern graphite mills and is a simple, cheap and compact machine, giving satisfactory results when properly handled. It works automatically and continuously and can be adjusted according to requirements.

This machine, see Fig. 23b, consists of a swinging door blower, B, with check valves to prevent the downward passage of air conveying rapid pulsations of air into the tubes T, one half inch wide, of sieve cloth, through the sides and tops of which it is discharged,

passing up through the bed of ore and effecting the separation between graphite and the gangue. These gauze tubes T are open at the end to the blower, to receive the wind and on the under side to prevent them from choking with fine ore. They are placed at



 $^{3}_{16}$ ,  $^{1}_{4}$ ,  $^{3}_{8}$ , or  $^{1}_{2}$  inch apart according to the grade of ore treated—the finer the nearer. The ore is fed in through a hopper H, passing under the adjustable gate G, forming a jigging bed and discharging clean graphite over the adjustable tail C. The tailings which completely fill the hutch below the tube T settle slowly and are

discharged by the regulated roller R. The swinging-door blower is actuated by a cam on the main shaft with six projections; the latter give the downward motion through an arm on the shaft with a spring, which gives it the quick upward pulsation, and an adjustable strap, which limits the amount of pulsation. Upon the cam is an adjustable crank pin, which serves as a pivot for a pawl acting upon a ratchet wheel to drive the discharge roller R.

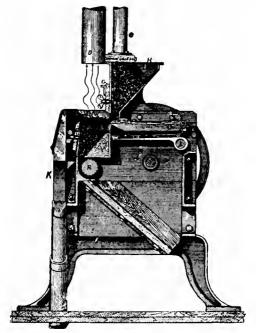


Fig. 23.—Krom Air Jig, as manufactured by the International Dry Concentrating Co. N.Y.

The roller therefore acts in concert with the blower. The width of the bed is four feet. The machine is run with 420 to 750 pulsations per minute. It treats from 300 to 600 pounds per hour, using one eghth horse power.

The coarsest size claimed as capable of being treated by this machine is six mesh and the finest size is 140 mesh. The machines are run in practice with two successive treatments, the roughers and the finishers; the roughers make for clean concentrates and the finishers for clean tailings. The apparatus, when carefully watched works satisfactorily, but it has no power of self

regulation. This causes the machine, if fed more rapidly than normal, to contaminate the concentrates the flakes with the tailings, and if fed less rapidly to lose concentrates in the tailings. And, further, if the feed be regular in quantity but the percentage of graphite variable, then the rise in percentage will enrich the tailings and the fall in percentage will contaminate the heads.

## THE HOOPER PNEUMATIC CONCENTRATOR.\*

The following are the essential features of this apparatus, which is illustrated in Fig. 24 and 25. Through the chamber A Fig. 25, runs a rectangular diaphragm (a). This diaphragm is composed of an outer rim of leather, the sides of which are firmly bolted

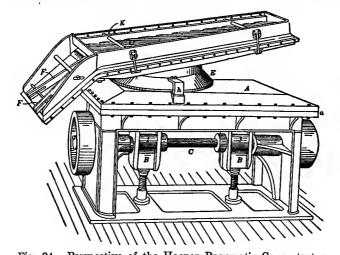


Fig. 24.—Perspective of the Hooper Pneumatic Concentrator.

between the upper and lower sections of the air chamber. Within the chamber the leather is firmly attached to a strong wooden frame (b), which is divided by transverse wooden braces (c). Between these braces and attached to them are two rubber flaps resting upon a sheet of perforated metal. The diaphragm is connected to two eccentric boxes B Fig. 24, in which revolve a fixed eccentric attached to the working shaft C, each eccentric being cased by a loose eccentric sleeve (d), which can be adjusted and held by a set screw (e), allowing a throw of one-eighth to one and

<sup>\*</sup> Richards, Ore Dressing, page 820.

a quarter inches. A movement is thus communicated to the diaphragm, which discharges at each revolution an air blast to the chamber A, which blast then passes through the fixed diaphragm G, also arranged with rubber flaps and is discharged through the grated sieve (g), upon a broad cloth bed (f), stretched over same. Resting upon the broad cloth bed is the concentrating top, which consists of two sets of guide strips, running diagonally to each other and at angles of from 30 to 45 degrees with the side of the frame.

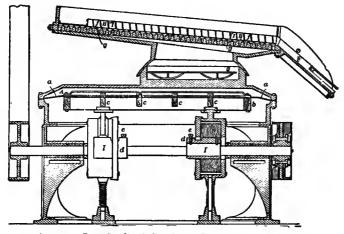


Fig. 25.—Longitudinal Section. Scale—3 inch—1 foot.

The lower set of strips H are of brass  $\frac{1}{8}$  inch thick,  $\frac{1}{8}$  to  $\frac{1}{4}$  inch high and  $\frac{5}{8}$  to  $1\frac{1}{4}$  inches apart, depending upon the material to be treated. The upper set of strips I, called skimmers run upon and diagonally across the lower sets. They are also of brass  $1\frac{1}{2}$  inches thick,  $3\frac{1}{2}$  inches high and  $\frac{5}{8}$  to  $\frac{3}{4}$  inches apart. These upper strips terminate two inches from the left or discharge side of the top for a distance of twenty-three inches from the discharge end, thus leaving a free discharge channel K for the gangue and tailings. The concentrating top may be removed from the concentrating bed at will. Any desired vertical or lateral inclination of the concentrating bed is obtained by means of a universal joint E, which is held in the desired position by means of two clamps attached at opposite sides of same, as shown at (h). The maximum inclination towards the discharge side is  $11^{\circ}$  and that towards the concentrating side  $5^{\circ}$ , depending upon the character of the

ore being treated and the mesh to which it has been sized. As a general rule the larger the mesh and the heavier the mineral, the greater the inclination in both directions. The crushed ore is fed from a hopper (not shown) placed at the head of the concentrating bed. This hopper is adjustable in position, and is provided with small sliding gates by means of which the flow is adjusted.

It will be evident from the foregoing, that when crushed ore composed of particles of different gravities, is fed upon the concentrating bed, the pulsations through the broad cloth, due to the blasts before described cause the heavier mineral particles to be thrown to the bottom, where they settle down between the lower metal strips and are thus guided in the opposite direction towards the tailing side of the table. After the bed is filled to an even depth of from \(\frac{1}{2}\) to \(\frac{3}{4}\) of an inch and the resulting products of concentrates, middlings and tailings begin to flow regularly and smoothly over the discharge end of the table, they are guided to any point of disposition by means of wooden guide strips F. It is found that the various minerals contained in an ore classify according to their specific gravities; the heavier mineral, being interrupted in its flow by the side of the concentrating top, is spread out in a well defined strip by the action of the upper skimmer, the next heaviest taking its place beside it, etc. is therefore a distinct separation of all the minerals, should there be sufficient variance in their specific gravity.

To obtain the best results the ores treated should be below 2mm, and should be closely sized, say through a 20-mesh screen on a 30-mesh, through 30 on 40, 40 on 60, 60 on 80, 80 on 120 and 120 on 250. The speed of the machine varies from 350 revolutions per minute in the case of coarse material to 450 for This variation in speed is obtained by means of cone The stroke or force of air is varied by the length of eccentric throw by adjusting the eccentric sleeves before described. The greater the throw of these eccentrics the stronger the air The heavier the material treated the heavier the air blast required. All machines are now supplied with an adjusting device, by means of which the throw of the eccentrics may be altered at will without stopping the machine. The capacity of the machine varies from 9 to 16 tons per day of 24 hours, according to the character of ore treated, and the horse power required varies from 11 to 2.

# SEPARATION BY FORCE OTHER THAN AIR BLAST.

In their simplest form these machines consist of a rotating distributor and a fixed receiver. The ore which must be absolutely dry and perfectly classified, although in the shape of fine sand, is fed into the distributor, which is so arranged that the particles of ore are projected from it with a uniform annular velocity, the particles being of approximately the same size, those of lower specific gravity are sooner overcome by atmospheric resistance and fall short and are collected in one compartment of the receiver, while those of higher specific gravity are thrown further and fall into a separate compartment. Upon these principles two appliances have been built, one the Pape-Hanneberg separator and the other the Clarkson-Stanfield patent centrifugal concentrator. Tests have been made with these machines on graphite ores containing to some extent metalliferous gangue, but the results are not known. It appears, however, that they are not well suited for ordinary ores, in which the gangue consists of the lighter materials, the similarity in the specific gravity of all the constituents of the ore excluding a separation by centrifugal force.

The Mumford and Moodie separator employs also the same principles as the machines just mentioned, but with the aid of an air blast. On account of its widespread employment both in America and in England for the treatment of various classes of ore, a description\* of the same is here given:—

Mumford and Moodie's Separator has a horizontal disc, D, revolving at high speed, see Fig. 26. Upon this, the dry ore to be separated is fed in a steady stream from the hopper C. The particles are thrown out radially in a horizontal direction, but are stopped by an enclosing vertical, truncated cone, expanding slightly downward, which surrounds the disc. In the annular space between the disc and the cone is an upward current of air induced by fan blades, A, revolving with the disc. This current lifts the lighter portion and discharges it in an outer chamber, E, while the heavier particles fall in an inner chamber, H. After having dropped its charge, the air returns from the outer to the inner, as shown by the arrows, being distributed to the pulp by the perforated plates, G, and acts over again. The machine is made in three sizes,  $3\frac{1}{2}$  to 6 feet in diameter, and these treat

<sup>\*</sup> Richards, Ore Dressing, page 818.

from 1 to 4 tons per hour. The advantage lies in its compactness for performing the duty of a screen, separating fine dust

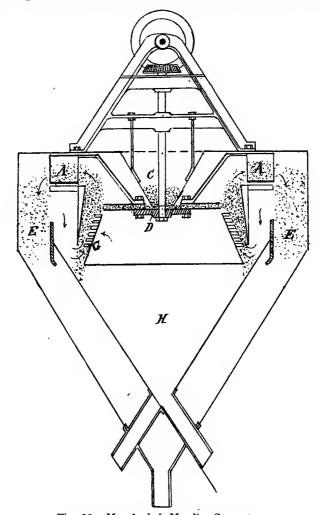


Fig. 26.—Mumford & Moodies Separator.

from coarser material. Two hundred are in use in England and America.

# APPARATUS USED IN THE WET METHOD.

A number of machines have been used for wet crushing of graphite ores, but it appears that only a few of them have been

employed with success. Amongst these may be mentioned, rolls, ball mills, the edge stone or Chili mill. In many mills stamps are being used, but these, as experience has shown, are very inefficient granulators because of their small screening surface and the inability of the ore to escape from the mortars, until reduced to the mesh of the screens or to excessive fineness. Their use in graphite concentration mills has therefore been generally abandoned. About the best work that stamps will do in crushing to 30 mesh is 60 pounds per horse power hour. Rolls on the other hand are thoroughly efficient granulators, they give approximately four times that output with the same expenditure of power; they can be used in wet crushing down to about 20 mesh with very good results and with fair results to 40 mesh, if there is not much talcose or clayey matter in the ore.

The ball mill is an efficient pulverizer. A well designed mill has a large screening area, and means are provided for the material under treatment to escape easily from the action of the balls and go over the screen, through which they will pass as soon as ground fine enough. The product is therefore granular in character and the power consumption is relatively low. The drawback is the high consumption of metal per ton of ore crushed, but this does not effect the advantages in other respects. The ball mill combines crushing, elevating and screening apparatus in a single machine. Its compactness renders it especially desirable when small units are required.

# EDGE STONE MILLS.

This mill, called very often the Chili mill, has vertical rollers running in a circular enclosure with a stone or iron bottom. The action combines grinding and abrasion with rolling or pressure. This mill is largely used in Austrian and Bavarian separation plants and, on account of its simple construction gives very little trouble, while the results obtained in pulverizing graphite ore are highly satisfactory. The capacity of this mill is not large compared with that of many other crushing machines but it is claimed, that better work is done. The centre of the roller is rolling upon the fragments while the margins are sliding upon them, the outer is sliding forward, the inner backward. An illustration of this mill is shown in Fig. 27. The two rollers have detachable rings made of rolled steel, and are rolling upon journals on a horizontal iron arm attached to and revolving with a vertical spindle. The journals for the rollers are generally capable of vertical adjustment

to compensate for the wear of the roller. Behind each roller are attached steel arms carrying wire brushes or scrapers to distribute or loosen the pulp. The mill is fed by shovel or by conveyor from the Blake crusher. It can be used either dry or wet, but

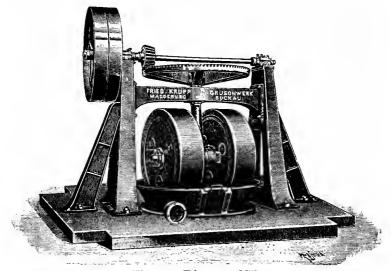


Fig. 27.—Edgestone Mill.

for the crushing of graphite ores, generally, the wet treatment is practised. In the latter case a screen is placed in the circular enclosure, through which the pulverized material is discharged. If it is desired that the latter shall have a certain degree of fineness, it is necessary to attach to the apparatus a revolving or pulsating screen, which separates the finer from the coarse. The latter is again sent back to the apparatus for regrinding.

The following table gives further particulars regarding size, capacity, etc., of an edge stone mill manufactured by the Krupp's at Magdeburg.

TABLE 38.

Diameter of runner Milli- meters.	Width of runner mm.	Revolutions per minute	Horse Power used	Capacity per hour Kilos	Weight of whole appara- tus	Weight of runner Kilos
500	125	45	0.5	35	850	165
$\frac{800}{1,000}$	200 250	30 25	$\begin{array}{c c} 2.00 \\ 4.00 \end{array}$	150 250	$2,950 \\ 4,100$	1,000
1,500	400	15	8.00	800	12,800	3,000
2,000	500	12	12.00	1,600	27,000	6,800

The construction of the Bryan roller mill is based upon the same principle as that of the mill just described, with the only difference that three rollers are used instead of one. A stationary cylindrical centre post stands up in the middle of the pan, to the top of which is keyed a branched support, which carries the boxes for the horizontal driving shaft. Below this is fastened the horizontal bevel gear, which receives the power from the pinion. Still lower is fastened the revolving table, which carries the three ball and socket journals of the rollers.

It is claimed that this mill is especially adapted for fine grinding preparatory to settling the fines.

### CLASSIFIERS.

These are appliances for subjecting the ground ore to the action of water under free settling conditions. They are divided into two classes:—

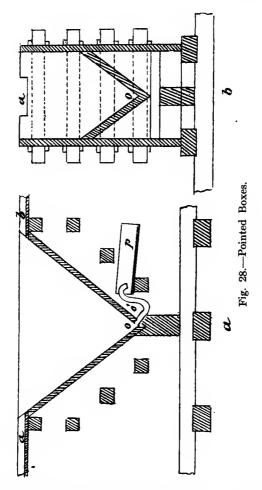
- 1.—Those, producing a series of products diminishing in size preparatory to subsequent treatment and,
- 2.—Those, which settle the whole material as completely as possible from water. To the first class belong all classifiers, to the second all settling apparatus and tanks. All these machines have a current of water, which carries away whatever grains may remain suspended in it. In the usual wet ore treatment, for example, in the case of heavy metalliferous ores, the designs and styles employed are very numerous, the value of each depends upon the character of the ore to be treated, but in the separation of graphite only a few of them can be employed with advantage, owing to the similarity of the specific gravities of the minerals, usually constituting the gangue.

To the group of classifiers belong the so called "Spitzkasten" or pointed boxes: In this separation the funnel boxes are rectangular pyramids, with the base upwards. They are designed for assorting sands direct from the rolls or the edge stone mill.

The material flows from the granulators through several of these boxes—each different in size and each delivering a different sized grain directly to the concentrators. In this way the sands may be separated into different degrees of fineness at very little expense, in a simple apparatus and without labor. These boxes have also the advantage of getting rid of surplus water, which would interfere with concentration. The first box is narrow,

which causes the water to flow swiftly, thus allowing only the coarser grains to sink, while the balance is carried into the next wider box, where the same quantity of water, spreading, assumes a slower motion, so that the next finer sand is separated and so on with the other boxes.

Fig. 28, (a) and (b) represent a pointed box. Fig. 28 (a) is a vertical longitudinal section, Fig. 28 (b) a vertical cross section. The



material flows from the stamps at (a) into the box. The sinking grains concentrate at the point (o) and flow out through the ascending conduit  $(o^i)$  into the trough (p). The finer material, which can

not resist the current of water is carried over the spout (b) into the next larger box. The conduit  $(o^i)$  ascends in order to counterbalance the water pressure inside the box.

To obtain all the advantages of this apparatus, theoretical and practical knowledge must be taken account of in construction. The width of the separate boxes is important. It depends upon the quantity of the material, which is intended to enter the box per second, and on the degree of coarseness and density of the grains in it. According to experience, the first box, by which the coarsest part of coarse ore is to be separated, should have 1-10 of a foot width to each cubic foot of stuff flowing per second. Each of the following three boxes has twice the width of the preceding. The depth of the boxes is given by the inclination, and the latter measured from the horizontal line is 50.

Another apparatus, which is used for the classification of sands is the "Spitzlutte," in principle a hydraulic classifier. This apparatus subjects the sand which comes from the mill to an upward current of water in a confined space, and the particles, which have sufficient weight to settle down through the current, can go out through a discharge spigot below, while those, which cannot do so, go over to the next pocket of the classifier where an upward stream of less force awaits them and so on to a third and fourth pocket. Since the speed of the current decreases in the successive boxes coarser particles settle in the first and finer particles in the latter.

Fig. 29 shows an adjustable spitzlutte. This apparatus consists of a box with a transverse V section with the sides (b) of the V sloping 60 degrees. Inside is a V shaped displacer or prism (c), which can be lowered and raised by set screw (d). The displacer is always centered in the V box, and between the sloping sides of the two parts will be left adjustable spaces or tubes for bringing in and for taking out the carrying current. The length of these two tubes for coarse stuff, according to Richards, must be about 914 millimeters; less is insufficient for good settling, more is unnecessary. The width may be 620 millimeters and the thickness will depend upon the size of grain it is desired to lift, and upon the quantity of water in the carrying current. An apparatus of the above width and design to treat 283 liters (10 cubic ft.) per minute will require a thickness of 61 millimeters of a speed of current of 125 millimeters per second. The outlet of the pipe (i) is regulated by mouth pieces, of which there must be several of different sizes. Once these are regulated, there is no other work to be done, except to watch the regular flow, as some accident may cause the conduit to become choked. To obviate such mis-

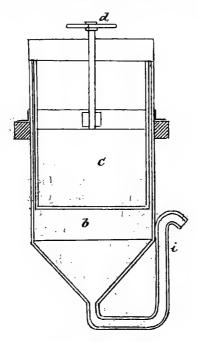


Fig. 29.—Spitzlutte.

haps the boxes are provided with a rod in the centre, on the lower end of which, just above the opening, are two small rings, so that if the sand or pulp accumulate by neglect of the watchmen this rod is turned until the stuff becomes loose enough to force itself through.

# BUDDLES.

These building tables or buddles, as they are generally called, serve to concentrate slimes and fine sediments on a circular bottom, inclined towards the periphery. The smaller grains of higher specific gravity are moved down the slope slowly by the water current or not at all, since they are in the lower slower current; while the larger grains of lower specific gravity are moved rapidly down the slope, since they project up into the upper rapid current, this action separates the waste from the values. The material

builds gradually up until about 10 or 12 inches deep has accumulated. The washing is then stopped and the produce cleaned out. The build ng up of the material is regulated by adjusting the tailboard, which prevents the ore from rolling off the table. Buddles must always be fed with classified products and when the feeding begins the material builds up and the sizing takes place upon a bed of more or less moving sand. After the building begins, the action continues uniform, so that the finer grains (heavy mineral) are deposited near the feed and the coarser grains (light material) at the tail. The finer particles nearer the head form a comparatively smooth surface, on which the large grains easily roll, while the coarser grains near the tail furnish a rough surface.

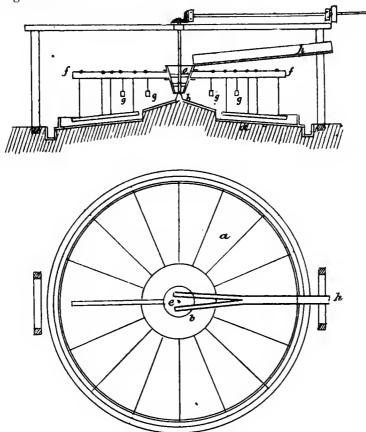


Fig. 30.-Circular Buddle.

The buddle is represented in Fig. 30. The conical bottom (a) is formed of wood and is 16 feet in diameter, sometimes 18 and 20 feet. On this the material is distributed; (b) is the cone supporting the feeding apparatus; (e) a funnel perforated with 4 holes and furnished at the top with an annular trough; (ff) are arms, carrying two brushes, balanced by the weights (qq); (h) is a launder for conducting the stuff into the funnel (e), from which it passes through the perforations, flows over the surface of the fixed cone (b), from thence towards the circumference, leaving in its progress the heavier portions of its constituents, while the surface is constantly swept smooth by means of the revolving brushes. By this means the particles of different densities will be found arranged in concentric circles. The arms usually make from 2½ to 4 revolutions per minute, and a machine 18 feet in diameter will work from 15 to 18 tons of material in ten hours. The feed cone (b) has a radius of three feet, 18 degrees slope and with its outer edge is 9 to 12 inches above the washing surface. A launder is placed around the buddle to carry off the waste water. When the buddle is set in operation the formation of the building cone is watched, and if the sand builds too fast at the upper end it shows that the pulp is too thick or that it is not fed in sufficient quantity. If the sand settles too thick below, it shows that the pulp is too thin or that it is fed in too great a quantity. As the bed fills up, plugs are inserted in the perforated tailboard. When the table is covered with the desired thickness of the pulp the different products are marked off in circles. Each concentric product is shovelled into its pile or bin or if waste to the waste launder.

In the case of graphite pulp the earthy or waste material settles down near the centre round the cone, while the graphite itself builds up near the periphery of the table. Generally three products are taken from the latter; waste, middlings and concentrates. The middlings are always treated a second time on another buddle or are charged with the original pulp coming from the "Spitzlutten" or "Spitzkasten."

### HORIZONTAL REVOLVING SIZING SIEVE.

A circular sizing sieve is sometimes used between the classifiers and film sizing tables for the extraction of graphite flakes. It has a diameter of from two and a half to three feet, is of convex shape and revolves round a vertical spindle. The sieve consists

of brass wire cloth or better of perforated galvanized iron, the mesh of which is determined by the size of the product treated. The construction of this apparatus may be seen from Fig. 31.

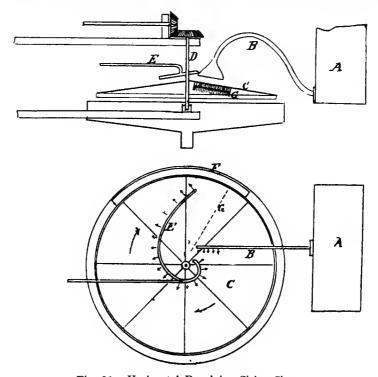


Fig. 31.—Horizontal Revolving Sizing Sieve.

B is the spigot pipe of a classifier A discharging the pulp on the sieve C, revolving in the manner indicated round a vertical spindle D; E is a spray pipe for conveying the flakes into a launder F.

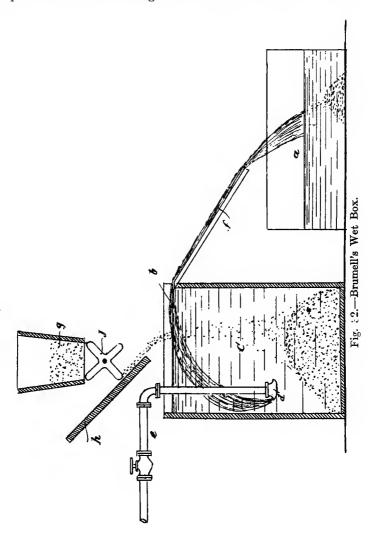
The action of the sieve is as follows:-

The pulp entering by B spreads out over the sieve and by the revolution of the table is carried before the fine water sprays of the pipe E. The smaller particles pass through the sieve in a launder underneath, while the concentrates consisting mostly of the larger flakes are swept before the fine water sprays into the launder F on the periphery of the sieve. In order to prevent the clogging of the latter a revolving brush G is placed under the sieve in the manner indicated, which cleans the holes from

clogging material, after they have passed the water sprays. The sieve makes from 4 to 6 revolutions per minute, depending upon the quantity and size of the grains.

## BRUMELL'S HYDRAULIC SEPARATOR.

This apparatus is being used in the mills of the Buckingham graphite district and has given excellent satisfaction. The in-



vention relates to the separation or concentration of ores, more particularly of graphite ores, and consists in feeding the disintegrated and dried ore upon a moving body of water, so that the minerals, which by their physical characters will float, shall be carried beyond those that will not float. The apparatus is illustrated in Fig. 32, which represents a vertical section of the same.

In the illustration, (a) indicates a receptacle for the graphite or other minerals, which may be carried to it by a broad flat stream or body (b) of water, thrown across the upper end of a receptacle (c), the sand or other heavier mineral substance not carried away by the stream dropping through it to the bottom.

The desired form of water stream is secured by the water issuing from a rose (d) on the end of the supply pipe (e) projecting down a short distance into the receptacle (c) and near one side of the same. The outlet side of the rose is turned upward and arranged to direct the water against the inside face of the receptacle.

The disintegrated and dried ore is fed to the stream through a hopper (g) and down a glancing board (h), a rotating, horizontal fluted roller or shaft (j) being located at the outlet of the hopper so as to secure a regular feed.

The process is distinguished from other processes by the fact, that the mineral is separated or concentrated when dry upon the surface, instead of wet in water, or dry in air.

## SETTLING TANKS.

These are used to settle finished products, whether concentrates or tailings from currents of water. They are of two kinds:—

- 1—Those which collect a great quantity of coarser heavier grains.
- 2—Those which take the overflow of the latter collecting all the fine grains. The tanks for the coarse material are as a rule much smaller than those for the fines and have in the Bohemian refineries the following dimensions:—From  $1\frac{1}{2}$  to  $2\frac{1}{2}$  meters long, 1 to  $1\frac{1}{2}$  meters wide and about 1 to  $1\frac{1}{4}$  meters deep. The collecting tanks in use are all of a rectangular shape and have frequently the following dimensions:—6 to 8 meters long,

1 to  $1\frac{1}{2}$  meters wide and  $1\frac{1}{2}$  meters deep. The graphite is allowed to settle in these tanks to a certain height, and then the water above the settlings is drawn off to waste, and the graphite slime shovelled out and transported to the filter presses. The best graphite comes from the last tank and the quality deteriorates from the last to the first tank.

Very often the settling tanks are arranged in series with short narrow connecting launders in such a way, that anyone may be cut out, emptied and flushed out through the bottom.

### FILTER PRESSES.

In the early days of graphite mining in Bohemia some 30 or 40 years ago much difficulty was experienced in freeing the slimes from the water preparatory to drying and grading. Many ways were thought of, and some of them put in practice; one method consisted of letting the slimes first dry in the air under long sheds and later dried over a fire. Another way was the application of a centrifugal dryer, but all these methods were slow and involved much manual labor, and could only be carried out successfully by taking small quantities at a time. Modern methods of production and refining on a large scale necessitated a more rapid way of separating the water from the solids, and this has led to the invention and introduction of the filter press.

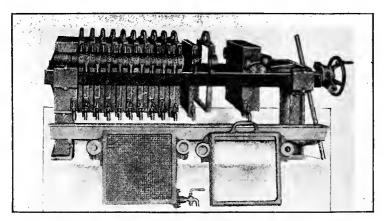


Fig. 33.—Filter Press.

A filter press, Fig. 33, primarly consists of a series of chambers formed either by recessed or by flush plates with frames between

them, the width of the latter determining the thickness of the cake. These plates and frames have a lug projecting from each side, which rests upon a pair of parallel bars. One end of each of these bars is secured to the front or the head of the press, the end plates and frames rest upon them and the back plate or follower is forced up against the frames by means of a heavy screw or hydraulic plunger, in a yoke or screw standard, to which the rear ends of the bars are secured. Over the surface of each plate or frame is stretched the filter medium consisting of cotton cloth.

The material to be filtered is pumped through a channel in the head of the press, and is distributed over the surface of the filter medium, the liquid passes through cored channels in the plates, the solid material being retained on the surface of the filter medium gradually filling the chamber.

The exit of the water is provided for by a drip cock at the side of each plate or is arranged to pass through a closed channel extending through the press. The former plan has the advantage of locating a defective cloth on the press, enabling that particular plate to be cut out by closing its drip cock, through which a cloudy solution would be passing. The closed channel on the other hand saves a little time in the operation of the press, there being no cocks to open and to close, each time the press is filled.

The slimes are pumped through the press under a pressure of from 90 to 100 pounds until solid cakes are formed inside the frames. The press is then opened by the hand screw or hydraulic plunger, the cakes removed one after the other from the frames, and after flushing all the channels and frames with a clear water current the operation can be repeated.

The pump used for charging these filter presses must be of special design, because any particle or grain that may get under the valves and stay there, may clog the pump and put same out of commission until the obstacle is removed, and these derangements may happen frequently. The pumps mostly in use in the Bohemian mines are the so-called "membrane" or "diaphragm" pumps; the plungers are separated in these from the valves by membranes of special construction or diaphragms, thus avoiding entirely the clogging of the apparatus. The slimes to be treated in the filter presses are generally collected in circular basins, where they are kept in motion by a stirrer. If the graphite has been treated chemically, as a rule after the process

of compression has been finished, a current of clear water, sometimes warm, is forced through the cakes in order to free the material from objectionable chemical solutions.

Filter presses are made in various designs and capacities, both in America and Europe; the writer saw, in several of the Bohemian and Moravian refineries, heavy presses treating from 600 to 1,000 lbs. of fine graphite at the time. Every 3 or 4 hours a press is discharged, so that in 12 hours from 1,800 to 2,400 lbs. of graphite is produced with one press. The moisture contained in the cakes varies from 18 to 23%; the latter are dried in ordinary dry kilns, which are kept at a temperature of 100 degrees centigrade for 24 to 30 hours; some of the finer products are then put up in barrels and sent to the market, while others are again pulverized in Ball mills or edge stone mills and then graded and shipped.

## THE MERRILL FILTER PRESS PROCESS.

A patent was granted on August 29th, 1905, to Mr. Charles W. Merrill of Lead, S.D., of U.S.A., for a new process of slime treatment.\* According to the specifications, the invention relates to improvements in processes for removing the semi-solid material from the containers of filter presses, and the primary object is to facilitate and cheapen the removal from the containers of the material, which will not pass through the filter medium.

A secondary object is to permit of the independent introduction of cleansing and precipitating of other material into the containers.

In the operation of filter presses or similar pressure filters it is customary to separate the units, of which each press is composed, and remove the solid, semi-solid or unfilterable material from the distance frame or container, usually separated by hand. This method is naturally expensive, in view of each unit of material so removed, as the wear and tear on the filtering medium, covering the filter plates is heavy, and the time so occupied during which the press cannot be used for filtering, greatly reduces its capacity per unit of time. Hence, the scope of the ordinary filter press is limited to the separation of solids from liquids in cases, where the value of such materials is such as to permit of the expenses outlined above. Furthermore, the necessity of removing the filtered material from each distance frame separately, results in the use of

<sup>\*</sup> Eng. & Min. Journal, 30 Sept., 1905, p. 602.

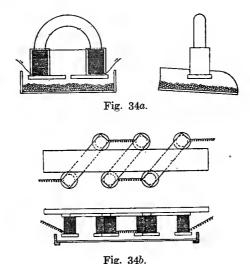
deep distance frames in order to increase their separate capacity. Moreover, the resultant thick cake of solid or semi-solid material increases the pressure necessary to accomplish the filtering; and thus adds another factor to the cost of operating said filter presses. The Merrill process consists in so constructing the containers of a filter press, so as to permit removing the solid or semi-solid material from the containers, without separating the units of the pressure filter. To accomplish this an inlet for each distance frame or container is provided, through which a liquid, vapor or gas is introduced under pressure, and the solid, semi-solid or unfilterable content is sluiced or forced out through an exit from the frame. This exit may be an independent outlet, or it may be the opening, through which the material to be filtered is originally introduced.

### ACCESSORIES FOR MILLS.

Among the many accessories for mills, may be mentioned the following more important ones.

- 1—Ore bins:—The varying production of ore in the mines calls for receiving bins large enough to serve for storage, when the mine is producing more ore than the mill can treat and to provide ore for the mill when none is received. It often happens that mining and milling are carried on for 24 hours, while hoisting and shipping cover only 12 hours. A common rule for the construction of bins is, that they shall hold at least 24 hours production of the mine, and they are often much larger. Intermediate bins are used in some mills to act as reservoirs, so that a temporary stoppage of one part of the mill will not necessitate the stoppage of the parts preceding and following.
- 2—Feeders:—In taking the rock from the breaker to charge fine crushing machines, the supply is regulated, but it often happens that a sudden rush of ore will choke them, and unless they are supplied with an extra amount of power and strength, which is generally not the case, they will break. To avoid this trouble feeders are used, which are kept constantly full of material, fed to the apparatus by automatic arrangement, mostly by oscillating gates.
- 3—Removal of pick-points, bolts and sticks:—To prevent strains on crushing rolls or clogging of different machines, coarse screens are sometimes used to remove pick-points, sticks, etc.,

from the ore after it has been through the breakers. For the extraction of iron in some mills powerful electro magnets are used. An illustration of two of the designs employed is given in Fig. 34a and b.



4—Dust-fans:—When the ore is treated dry, there may be so much floating dust emanating from the crushers, pulverizing mills and screens, that fans are needed to remove it. These suction fans blow the dust generally into a bag room, which is a simple and effective means of catching the same. This consists of a room, in which a large number of burlap or cotton cloth bags are suspended. The air passes through the meshes of the bags, while the dust is retained in the latter.

5—Screens and sieves:—These are divided into two classes: shaking and revolving screens. The first are often used in the separation of graphite flakes from the dust, a revolving brush below the screen being used to free the holes from flakes which may clog them. They may receive an endwise, sidewise, up and down or gyrating motion, the number of pulsations being between 200 and 300 per minute. The revolving screens or trommels may be divided into cylinder and prisms or cones. The cone sieves empty themselves, and the cylindrical and prismatic sieves have an inclination for the discharge of the oversize. The revolutions range from 16 to 20 per minute. The surface of the sieves are

made of various materials, the coarser ones of strong wire cloth, the finer or grading screens of plated steel wire, brass or silk bolting cloth.

6—Slime pumps:—Centrifugal pumps are more largely used than any other form for elevating slimes, because they have no valves or plungers to be injured by the grit. They consist generally of a set of fans or blades, carried on a revolving shaft, surrounded by a heavy casing, and their action is exactly the same as that of dust fans. The pulp enters the casing round the shaft and is carried by centrifugal force to a discharge opening. These pumps are very simple in construction, they deliver large quantities, are of low cost and require but little attention. The most common sizes used in the mills are those having 2 or 3 inch discharge pipes.

# SUMMARY OF PRINCIPLES IN THE SEPARA-TION OF GRAPHITES.

Having described individually the various principal machines used in graphite mills, there remains now the consideration of the mill as a whole, including the various combinations of principles and the different arrangements of the apparatus employed. But the states of mineral aggregation existing in graphite ores occur in such varieties, that it is difficult even impossible to generalize on the subject under consideration.

Two ores of even identically the same composition, sometimes cannot be treated by the same method, on account of the different physical character in which the mineral occurs. This difficulty is apparent in the case of ores containing only flake graphite, and in those containing only dense or amorphous graphite; both of these ores occur in some localities in gneiss, but it would be futile to attempt to apply the same process, which gives good results in the separation of one, to the separation of the other from the gangue. The case is even more complicated for ores containing both varieties.

Another difficulty arises from the fact that in some localities the gangue, accompanying the mineral, changes in the mine as development proceeds, and it must be said that this is one of the principal sources of difficulties in the construction, operation and adjustment of graphite mills. The selection of one or the other method is very often based on chances and with very little regard to what the general "run of mine" is likely to be. When a deposit of graphite is discovered, as a rule a number of test holes are sunk to determine the character of the ore, or to trace the ore lode on the surface; where the ore appears to be of satisfactory quality and quantity, larger excavations are made or a shaft is sunk or a tunnel is run, following the trend of the ore body, and in the majority of cases, where quick returns are essential for the continuation of the enterprise, the construction of a mill for the treatment of the ore is proceeded with, based on preliminary tests with ore generally taken from chutes, which promise to deliver the bulk of the material to be treated. Very often, however, it is found in the further development of the mine, especially in the crystalline formation, that the ore changes in character, both physically and mineralogically, that, for instance, an ore originally flaky turns into a combined flaky and amorphous condition, that, further, the gangue with the accessory minerals changes and so on, and the result is that the mill which was originally laid out and constructed to suit a specific class of ore has now to face entirely new conditions. A series of experiments then demonstrates, that the arrangement of the apparatus and perhaps the latter themselves also have to be changed, in order to meet these new conditions, sometimes the entire system has to be abolished and has to be replaced by a new one, which in the majority of cases can only be effected by the expenditure of a large amount of money.

The writer has a case in mind where a property was superficially tested and found to contain flake graphite in quartz. Before proceeding with the development of the mine, to determine the extent of the ore body and its character, a mill for the treatment of the ore was erected. In developing this mine, however, it was discovered that the contents of flake diminished, and that instead amorphous graphite appeared; that further the gangue changed from quartz into pure calcite. The consequences are obvious. Changes after changes had to be made in the mill in order to meet the new conditions, arising solely out of the development of the mine, but with little success, until it seemed inadvisable to alter further the existing milling process. The construction of an entirely new mill was the only remedy, but the financial resources being exhausted, mill and mine were shut down.

We learn from this example, that the construction of a mill at a mine can only be proceeded with when all the conditions in the mine, the character of the ore, as well as the extent of the ore body, is fully known, and this can only be attained if the mine is properly developed, the stopes are laid open and the different ore chutes thoroughly tested upon their contents as to useful and waste material. Until such is the case experiments on a large scale with the ore should be deferred, the preliminary outlay for opening up the mine is insignificant compared with the large expenditure, to say nothing of the trouble and annoyance, resulting from the premature erection of a milling plant, whose method is based on chances, hasty judgment and insufficient knowledge of the conditions really existing in the mine.

Mechanical ore separation is an ancient art, but during the last ten years it has experienced some remarkable improvements, which have revolutionized the practice of ore dressing in general; it appears probable that there will be still further innovations of a character not yet clearly foreseen, except by some inventors who are working on special lines. Even as recently as five years ago there were practically no methods of mechanical separation except hand sorting, jigging and washing on tables and magnetic separation, the last method having only a very limited application. Since then radically new processes, like electrostatic separation, oil concentration, and the flotation of minerals in certain baths, have been invented. In these ways it has become possible to separate a wide range of minerals of close specific gravity, that cannot be separated by jigging or slime washing. In this way it has been demonstrated that phosphate can be separated from hornblende, the difference in specific gravity being only 0.35; further molybdenite, monazite, and other rare minerals can be separated from their gangue, which frequently it is difficult to do by any ordinary gravity concentration. Experiments have shown that graphite can be separated electrostatically from its gangue, and it is not unlikely that such a process will before long be developed commercially. The experiments have been to a certain degree successful, and it may only be a question of time and of some be of interest to quote the following note from W. R. Ingalls,\* on general ore separation: "Probably few realize the probable extent of development, which they will experience during the next ten years, or what results may be achieved by them. It is quite certain that no single process is a universal panacea for all the

<sup>\*</sup> Eng. & Mining Journal, 1905, page 643.

difficult problems of ore treatment. For certain ores one process is best adapted; for other ores another process. For still other ores the best results may be achieved by a combination of two or three, or even more of the special processes. This is a direction of effort that has not yet received very much attention. It will be however, the logical result when the design of such ore dressing plants passes out of the hands of the promoters of special processes into those of the engineer, who is retained by his clients to secure the best results."

## Dry and Wet Methods Compared.

In the separation of the graphite from its gangue the physical qualities of the mineral allow the use of special processes. On account of its soft character it can be more readily crushed and taking its low specific gravity into consideration a separation is made possible by disintegration and subsequent settling. The quality of certain kinds of graphite to deliver a large quantity of flakes or lamellæ, while the gangue breaks up in rounded lumps or particles, allows a separation of the flakes from the gangue by screening.

As to the wet method the same can only be successfully applied when the mineral is treated with an abundance of water, and to get rid of the latter, especially where large quantities are handled, is a matter of great importance and a source of large expenditure. The employment of filter presses with subsequent drying are operations which form an essential part of the whole separation scheme and much attention has to be paid to this particular work if expenses shall be kept down.

A further disadvantage in the wet method lies in the great losses in the tailings and in the difficulty in settling the slimes; further the greasy flotation of the mineral causes some loss and sometimes much trouble. In countries with severe winter seasons like Canada much difficulty is experienced from freezing in cold weather.

On the other hand a great advantage in water separation is derived from the fact that it disintegrates earthy and clayey matter, freeing the particles cemented together by the latter; the particles become free for individual treatment. Air affects neither of these results.

As to the dry method, the chief claim in favor of the same is the employment of a cheap medium of separation, that of air, thus doing away with the installation of special pumping machinery, the cost of pumping of the slimes and the drying of the finer products. In the case of an amorphous graphite or of a flake graphite rich enough in the amorphous variety, it is easy to draw off by fans the graphite dust emanating from the various pulverizing and screening apparatus and this dust in the majority of cases is rich enough to be shipped direct from the collecting chamber. It gives no trouble from freezing in cold climates and the resulting products are dry and ready to be shipped at any time. On the other hand it must be said that the treatment with air requires the whole of the ore to be made perfectly dry at the start, which in the case of wet mines is also a costly operation.

In water concentration graded crushing is advantageous, since the coarser sizes and the dust are easily treated; the reverse is done in the case of air, which cannot be employed for coarse sizes, and the fine crushing makes dust which cannot be as well treated as by water.

## Specific Gravities of Graphite and other Minerals.

In the following table \* are summarized the specific gravities of a number of minerals, which either may constitute the gangue in the ore or occur occasionally in the latter:—

	Chemical Composition.	Spec. Gravity.
Albertite		1.097—
Albite	NaAlSi <sub>3</sub> O <sub>8</sub>	2.62 - 2.65
Anhydrite	CaSO <sub>4</sub>	
Apatite	$ 3Ca_3P_2O_8+CaF_2CaCl_2$	3.19 -3.23
Aragonite	$CaCO_3$	2.93 - 2.95
	As	
Arsenopyrite	FeAsS	5.9 - 6.2
Barite	BaSO <sub>4</sub>	4.3 —4.6
Bauxite	Al <sub>2</sub> O <sub>.3</sub> 2H <sub>2</sub> O	2.55
Biotite	1	2.7 —3.1
Calcite	CaCO <sub>3</sub>	2 713
Celestite	SrSO4	3.95 - 3.97
Cerussite	PbCO <sub>3</sub>	6.46 - 6.574
	Cu <sub>2</sub> S	
Chalcopyrite	CuFeS <sub>2</sub>	4.1 -4.3
	$Al_2O_3$	
Dolomite	(Ca, Mg) CO <sub>2</sub>	2.8 - 2.9
Fluorite	Ca F <sub>2</sub>	3.01 -3.25

TABLE 39.

<sup>\*</sup> Dana, Mineralogy.

	Chemical Composition.	Spec. Gravity
Feldspar	PbS	2.47 -2.67
Galena	$\dots    ext{PbS} \dots \dots \dots$	7.428
Garnet	$R_2R_2$ (SiO <sub>4</sub> ) <sub>3</sub>	3.15 - 4.3
Graphite	C	2.09 - 2.23
dypsum	$\operatorname{CaSO_4} + 2\operatorname{H_2O} \dots$	2.31 - 2.33
	$\operatorname{Fe_2O_3}$	
	$2H_2O$ , $Al_2O_3$ $2SiO_2$	
Limonite.	$2 \mathrm{Fe_2O_2}$ , $3 \mathrm{H_2O}$	3.6 - 4.0
Magnesite	MgCO <sub>3</sub>	3.00 - 3.12
	FeO Fe <sub>2</sub> O <sub>3</sub> $$	
	Mn.,	
	$M_0S_2$	
	$H_2KAl_2(SiO_4)_3$	
	$SiO_{2}nH_{2}O.$	
Pyrite	$\operatorname{FeS}_2 \dots$	4.95 - 5.10
Pyrolusite.	$Mn\tilde{O}_2$	4.82
Pyroxene	$RSiO_3$	3.2 -3.6
Pyrrhotite	$\operatorname{Fe}_{11}\operatorname{S}_{12}$	4.58 - 4.64
Duartz	$\operatorname{SiO}_2$ .	2.65 - 2.66
	CaWO4	
	H <sub>4</sub> Mg <sub>3</sub> Si <sub>2</sub> O <sub>9</sub>	
	$Sb_2S_3$	
	$$ $SrCO_3$ $$	
	BaCO <sub>3</sub>	
Wallastonite		
	ZrSiO <sub>4</sub> .	

The chief difficulty with the concentration of graphite arises from the intimate association of the constituent minerals with each other, and from the similarity of their specific gravities; indeed, it is not too much to say, that the proper separation of the gangue from graphite offers one of the most intricate problems in modern ore dressing. Both the dry and the wet methods have their great faults, but experience seems to point to the wet method as the more successful.

In the Bohemian and Moravian mines, where most of the graphite ore has a uniform character, the present practice of refining is the concrete result of the experience of many men, extending over a period of some 50 years, which has naturally evolved a system of separation, that conforms to the peculiar conditions of those districts. Here the wet treatment is found to be best suited to the peculiar conditions of the ore, further improvements in that process are still being made, but they are much more likely to originate in the districts themselves than outside of them. In the wet method, as carried out in the Bo-

hemian and Moravian mines, it appears, as investigations have demonstrated, that the coarser aggregates accompanying the graphite can be separated from the latter in a satisfactory manner, but substances which are intimately associated with the mineral in a very fine state of division, for instance oxide of iron and the silicates cannot be separated by a current of water. In graphite ores containing these substances, the contents of ash cannot be reduced by the wet method as demonstrated by Stingl, who has made a number of tests in his laboratory with different graphite ores from the Bohemian mines. In this connection it may be interesting to note that already Schaeffel\* in 1866 made a series of tests and investigations with wet refining of graphite in the laboratory of the K.K. Geol. Reichsanstalt at Vienna. Schaeffel demonstrated in a conclusive manner that with the washing method, nothing else can be obtained than the separation of the graphite from the coarser aggregates, generally accompanying the mineral. The ash of the graphite, that is the quantity of those non-combustible substances, which are intimately associated with the mineral in an extremely fine state of division cannot be reduced by the most intricate washing processes. Stingl says further, that the efforts which have been made in that direction in the Austrian works have met with little success. A reduction of the ash can only be obtained by chemical processes, but in order to do this the chemical constitution of the ash itself must be known, this however changes a great deal in one and the same mineral. Whether substantial improvements in the washing methods have been made in Austria in the last few years to reach the purpose under consideration is not known. as authoritative statements on that subject are not available, but it is well known that for the finer purposes of manufacture chemical processes are employed for refining the mineral in some of the larger Bohemian and Bavarian works.

The selection of the most profitable milling process for a given ore, that is a process that delivers a comparatively clean product, without leaving at the same time much useful material in the tailings, is a matter of the greatest importance for a graphite mining enterprise; but as the ores as outlined in Chapter IV are of a complex character it is not possible to lay down exact rules, nor can they be of such nature as to cover all cases. If, therefore, in the following an attempt is made to give a general

<sup>\*</sup> Jarhbuch, K. K. Geol. Reichsanstalt, 1866, 126.

outline of the wet and dry methods in use or of combinations of both, as practised in various countries, it must be fully understood, that although theoretically the principles introduced would allow a perfect separation of all the graphite from the gangue, practically such is very difficult to accomplish and is rarely obtained. Indeed it will be found that some of the processes considered, especially as far as the treatment of tailings is concerned, lack completeness and details, but this is principally due to the difficulties encountered in the attempt to study the mill schemes on the spot, as applied to certain classes of ore and also to the very meagre literature on the subject. Graphite mill owners and operators as a rule do not like to divulge the r secrets in ore dressing; they guard these jealously and in consequence nothing is published, and in most cases a request for permission to inspect their milling plants is refused. The reader will therefore understand the difficulties in collecting material, and that to produce a comparative treatise on the subject is not an easy task. Moreover, the separation of graphite is being conducted in so great a variety of ways, to meet so many conditions, that it is quite impossible to formulate any specific rules of operation, which will apply in all cases. Indeed it would be very hard to find any two mills constructed alike, or operated in the same manner.

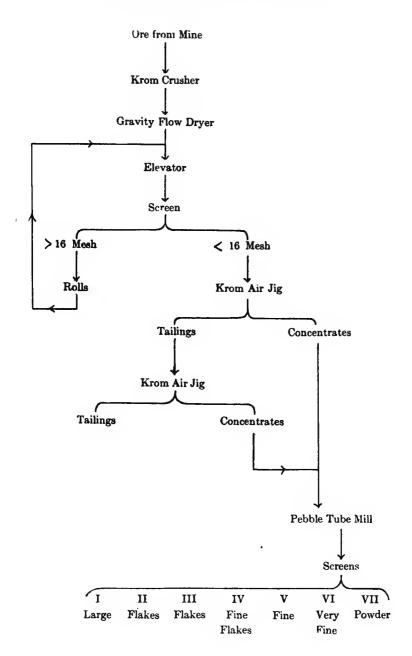
In order to illustrate the working of the dry concentration method, as adopted in some of the American plants, a section and plan of a mill are given in Plates XVI and XVII.

In the following the general outlines are given of milling processes, which are in actual use in North America and in Europe. It will be noticed that the initial stages of the operations are in principle the same, namely, gradual comminution of the ore by a series of machines; the finer the ore is to be crushed the more numerous are the members of the series, except that in the case of the ball crusher, it is possible to effect the reduction satisfactorily with only two machines, a rock breaker and a ball mill regardless of the degree.

## MILL SCHEME I (DRY).

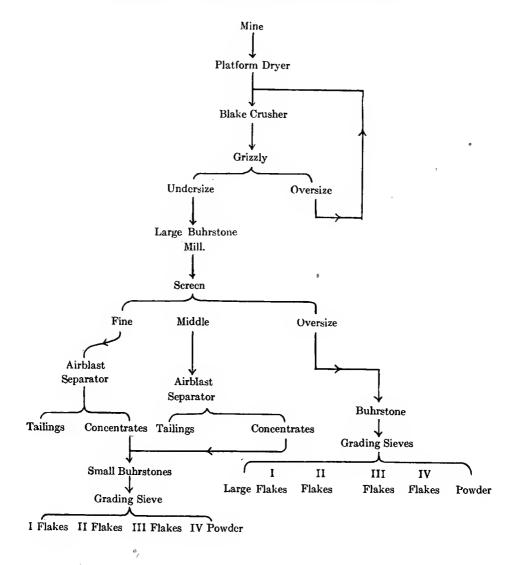
Character of ore: Desseminated graphite in crystalline limestone and quartz.

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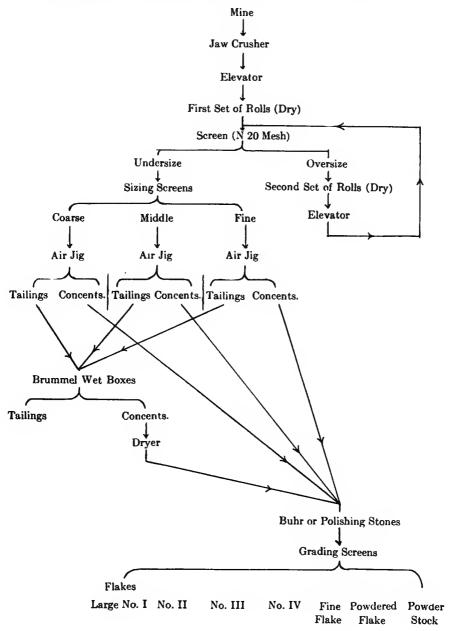
## MILL SCHEME II (DRY).

Character of ore: The graphite occurs in hard scaly particles, large and small in a matrix of gneiss, feldspar and quartz, with iron pyrites. The ore is soft and partly decomposed.



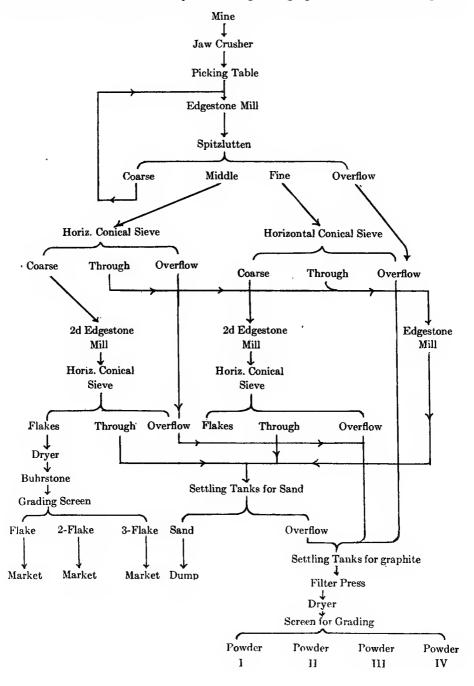
## MILL SCHEME III (WET & DRY).

Character of ore: Flake graphite in a matrix of hard gneiss.



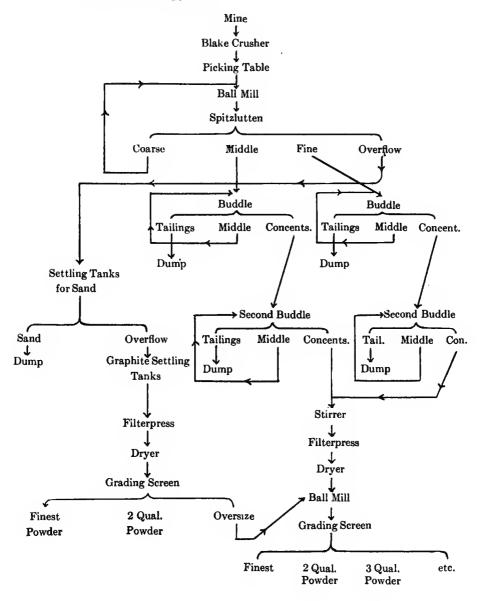
## MILL SCHEME IV (WET).

Character of ore: Contained flaky and amorphous graphite in a matrix of quartz.



## MILL SCHEME V (WET)'

Character of ore: Amorphous graphite in a matrix of quartz with some iron pyrites.



A graphite washing process\* which is employed in one of the Austrian mines, acknowledged to be the largest graphite producer in the world works as follows:—

The ore occurs in gneiss and consists of graphite, kaolin, calcite, quartz and pyrite; it is ground to fine pulp in two edge stone mills and then passed over six settling tanks for sand each 1½ meters long, 1 meter wide, and one meter deep. The gangue settles in these and is removed periodically. The overflow passes on to 18 settling tanks for graphite, each six meters long, 1 meter wide and 1½ meters deep. The graphite is allowed to accumulate in these to a certain height, and then the water above the settlings is drawn off after a stay of 48 hours and all the graphite slime is discharged to the filter press. The best graphite comes from the last tank and the quality deteriorates from the last tank to the Usually only three grades of products are made, so that the products of several neighbouring tanks are thoroughly mixed together before going to the filter presses. The slime is pumped through the big presses under a pressure of 6 atmospheres (88 lbs. per square inch) and each press yields in twelve hours 1800 to 2400 kilos of graphite in cakes containing 20% of moisture. A press is discharged every 3 or 4 hours. The cakes are dried at 90° to 100° C for 24 to 30 hours and then crushed dry in edge stone mills and sized for the market. In southern Bohemia the best grades for use by this method contain from 80 to 95% carbon, while in Moravia, where the ores are of poorer quality, 55% carbon is quoted for the best grade.

At the mill of the Philadelphia Graphite Company at Chester Springs, Penn., the graphite ore is crushed in rolls and then concentrated in log washers. The concentrated graphite is again ground in rolls and prepared for the market by air blast and sizing screens. The average rock is said to contain 28% graphite, but rock with 10% can be treated with a profit.

The process of graphite dressing in Alabama is patterned after the Austrian mills; this is fine crushing followed by concentration in settling tanks.

We learn from the above mill schemes that the arrangement of the apparatus in the mills is manifold, depending primarily upon the character of the ore, local conditions and objects in view, from which new combinations may suggest themselves. In this

<sup>\*</sup> Berg. & Huttenmannisches Jahrbuch, Vol. 42, page 95, also Richards Ore Dressing, page 1075.

connection it may be interesting to note that recent tests with flake graphite ore have been made on the well known Wilfley table with alterations of the tail board, and the writer has seen some of the products which seem to indicate its advantageous employment. The writer is, however, not aware that any of these tables are in use in graphite mills, but the reputation which the table has gained in the successful dressing of other ores should pave its way for its adoption for dressing graphite ore, even if this be only experimental at the start. It has been demonstrated that the Wilfley table has a very wide range of applicability and it has been proved capable of taking pulp, sized only within wide limits, and effecting therefrom not only a clean separation, but also a higher percentage of recovery than with the ordinary apparatus in use.

As to the general results obtained in milling graphite it must be stated that the loss in tailings is, in the majority of cases, large owing as stated before to the similarity of the specific gravities of the various constituents in the ore, which render a complete separation of the mineral from the gangue extremely difficult if not impossible. The losses in tailings in some of the Bavarian mills, which the writer has visited, are sometimes as high as 40%, and one mill in the United States is known to save only 50% of the graphite mined. In the majority of cases, it may be stated that the mills lose between 15% and 30% in the tailings. In one of the Passau mines the best ore contains 53.8% graphite and the best concentrates 89.2% graphite. The tailings vary from 22.3% graphite in the poorest to 36.8% in the richest.

## CHEMICAL REFINING OF GRAPHITE.

In the foregoing paragraph on mechanical separation it was pointed out that a reduction of the ash in graphite ores, in a closer sense those incombustible substances, which are intimately associated with the graphitic carbon in a very fine state of division, as oxide of iron and the silicates, can only be effected by chemical treatment. But in order to arrive at a rational method of chemical extraction, it is necessary that the constituents of the ash be known, but as the latter changes frequently, this aggravates the difficulty of successful treatment. By combustion of different samples of graphite in oxygen sufficient quantities of the incombustible substances may be procured and this investigated as to what reagents will bring it into solution. A procedure, con-

ducted in this manner, gives all the means for the determination of the necessary quantities of the reagents to be employed for the liberation of the graphite from the ash. Dr. Donath\* has made in this way a number of tests with ½ lb. of each of graphite ores and by a treatment with hydrochloric acid, caustic soda, heating with soda with subsequent washing with hot water, graphites even with a low percentage of carbon, (containing from 30% to 40% solid ash) can be raised to 97–98 per cent. carbon; he states further that with these simple agencies the same results can be obtained as by a treatment with more complicated processes (like the treatment with chlorine and fluorine, etc.)

If the non-combustible residue contains much oxide of iron, the latter can be completely eliminated by heating the graphite and then treating it with a diluted acid. By the contact of the carbon in the graphite with the oxides of iron in this treatment the latter are reduced to metal, which naturally can be completely dissolved with diluted acids without any difficulty. This dissolution takes place under strong liberation of hydrocarbons, as iron carbide will be formed. If the non-combustible residue consists essentially of silicates, which cannot be dissolved by hydrochloric acid, it appears advantageous to make a thick paste of the graphite and a concentrated solution of sodium carbonate and after drying the mass, to expose the latter to a red heat in crucibles. The heated mass is washed with hot water, then treated with diluted hydrochloric or sulphuric acid, and if much silica is present, finally with a solution of caustic soda.

Schoeffel found no graphites, whose non-combustible residue could not be completely extracted either by the application of one or by the successive employment of several of these agencies. Of course it would not pay to apply this method on a large scale to low grade graphites, containing from 30% to 40% ash, but graphites with carbon ranging from 80% to 85%, as for instance in a number of ores in Austria, can be purified with advantage by these processes and changed into products, which are equal to the best natural varieties.

These tests demonstrate, that it is possible to extract with fairly cheap chemicals the ash from nearly all graphites, but which of the above mentioned processes and what quantities of the reagents are necessary to secure the best extraction of the ash must be determined in each case by a quantitative and qualitative

<sup>\*</sup> Ibid, page 86.

analysis of the latter. These data furnish the basis for the application of the various processes in an economic way. It must be added, however, that it is not so much the variable contents of the ash as a whole as the chemical constitution of the latter, which excludes the establishment of certain rules for the application of one or the other process.

Well known is the method of Brodie,\* which is less adapted to a commercially economic cleaning of the impure graphites commonly occurring in nature than for the production of absolutely pure graphite in an extremely fine state of division. The roughly powdered graphite, freed as much as possible from its impurities, is mixed with the fourteenth part of its weight of chlorate of potash. This mixture is placed in an iron vessel: concentrated sulphuric acid (specific gravity 1.8) of double the weight of that of graphite is added and the whole thoroughly stirred and heated in a water bath, until no more gases escape: after cooling, the mass is put into water and thoroughly washed. The dry graphite is then exposed in a crucible to a red heat; it swells up considerably and is converted into an exceedingly fine powder. In order to clean the graphite thoroughly, the powder is then subjected to washing and settling in water. This method is specially adapted for the large scaly graphite of Ceylon. the graphite contains silicates and is to be made applicable to the manufacture of pencils, it is necessary to add to the mixture of sulphuric acid, chlorate of potash and graphite, a little sodium fluoride: the silicic acid escapes then as fluoride of silicon. the method of Brodie has several faults, for according to Gottschalk, if the graphite is heated with a mixture of sulphuric acid and an oxidizing agent it is liable to become oxidized.

Pritchard cleans the graphite in the following manner; which is applicable only to ore containing small quantities of ash. 18 parts by weight graphite, one part by weight chlorate of potash is taken and 36 parts of sulphuric acid (specific gravity 1.8). The whole is gently heated until no more chlorine escapes, the surplus of sulphuric acid is poured out, and a small quantity of sodium fluoride is added to the graphite paste. Finally the mass is thoroughly washed and the graphite exposed to a red heat, when it forms a red spongy mass.

Brockadon† cleans the finely pulverized graphite by fusion

<sup>\*</sup> Brodie, Polyt. Journal, 139, 215, 166 and 390 and Donath Ibid, 87. † Fehling, Handb. d, Chemie 3, page 505.

with sodium carbonate and subsequent washing with water, then with hydrochloric acid and again with water. The fine powder obtained after drying is moistened and compressed in forms under high pressure. The pieces thus produced have the appearance, hardness and density of the natural graphites; their conductivity for electricity is, according to Mathieson, 18 times greater than that of the natural graphites.

According to Winkler, to the finely pulverized graphite is added the same or double weight (according to its degree of purity), of a mixture of equal parts of carbonate of soda and sulphur, and the mass exposed to a gentle heat, untilthe blue sulphur flame, which burns in the beginning under the cover of the crucible, has disappeared. The slightly slagged mass after cooling is boiled with water and washed by continued shaking; the residue is treated with dilute hydrochloric acid, which brings all the iron into solution. During this treatment the graphite enters into a state of very fine division and takes much time to settle. The graphite, obtained in this way, leaves after washing only a very small residue of snow-white silicic acid which can be extracted, if so desired, by boiling with caustic soda.

According to another method, the oxide of iron should be reduced in closed retorts, whereupon it can be easily extracted by hydrochloric acid. It must be mentioned, however, that the reduction of oxide of iron takes place at the expense of the carbon of the graphite, and for this reason considerable loss of valuable substance may be expected; moreover, the silicates are not removed to any extent.

Bessel Bros., of Dresden, Saxony, use still another method\* for the refining of graphite on a large scale. The process works out as follows: In order to clean crude impure graphite, the latter is mixed with 1 to 10% of an organic substance and then heated with water until boiling begins. The graphite rises to the top of the liquid and can be taken off with large dripping spoons. A condition is that the organic substance, if liquid, is not soluble in water, and if solid is not impregnated with the latter. Preference is given to the employment of the following organic substances: all fats from animals and plants, all etheric oils, all resinous matters from plants and minerals, crude petroleum, paraffin, benzine, fusel oil, ozokerit, etc. Later (1887) Bessel Bros. have changed their original methods to some extent. According to this new method,

<sup>\*</sup> Deutsches Reichs Patent No. 42.

the graphite is intimately mixed with organic substances insoluble in water, particularly hydrocarbons in solid or liquid form, fusel oils and similar products. Warm water of 30° to 40° C. is added and the whole thoroughly agitated; a current of gas is then produced in the mass by the addition of other chemicals, and the gas bubbles cause the graphite flakes to rise to the surface of the liquid, while the gangue remains at the bottom. The current of gas is generally produced in the graphite mass by adding to the mixture of graphite hydrocarbons and water, some carbonates like chalk or metals which evolve with diluted acid carbonic acid gas or hydrogen and a diluted acid.\*

Bessel Bros. are reported as treating in this way large quantities of graphite ores coming from their mines at Kropfmuehle, near Passau. The graphite is an excellent crucible graphite; in the crude state it contains only from 25% to 50% of carbon, but by subjecting the same to chemical treatment it is raised to a high degree of purity to (92% and 94% of carbon).

E. B. Kirby has patented (U.S. 809,959, Jan. 16, 1906), an oil flotation process, wherein the pulverized mineral is mixed with a considerable quantity of water and with a substance immiscible with, but lighter than water, a solution of bitumen in kerosene, which in presence of water will adhere to some of the mineral particles, but not to others. The mass is violently agitated and then allowed to settle, whereby the particles, which have become coated with the bitumen solution, rise to the surface, this separation being assisted by gentle agitation and by blowing in a current of gas. The layer of floating matter is removed and washed, and the mineral particles are separated by filtration and then heated to recover the light hydrocarbons.

H. Putz† has made a number of experiments with graphite flakes and crude petroleum, and proceeded as follows:

The graphite is pressed and disintegrated between the hands, and 10 grams of the same is placed in an Erlenmeyer flask. Ordinary petroleum is added until a thin paste is produced, and the flask is a little more than half filled with water. After the whole has been well shaken for several minutes, the flask is completely filled with water. It will then be seen that the graphite forms on the surface with the petroleum a thin film, while the clayey and sandy substances sink to the bottom. After some time of rest the flask

<sup>\*</sup> Deutsches Reichs Patent No 39369.

<sup>†</sup> Jahresbuch fur chemische Technologie, 1886.

is carefully decanted, and with the addition of a little water the thin graphite film is allowed to overflow; the latter is subsequently washed with water and dried. The graphites before treatment had the following percentage compositions:—

TABLE 40.

Kinds of Graphite.	Water.	Carbon.	Ash.
Pfaffenreuth	3.28	53.78	42.95
Kropfmuehle	5.02	31.72	63.26
Germanndsorf (brown earth)	3.45	34.54	62.01
Kropfmuehle (good earth)	3.66	54.49	41.85
Kropfmuehle (brown earth)	2.88	36.22	61.80
Stierweide (near Germanndsorf) (black			•
earth).	2.70	54.25	52.05

The graphite earths of Pfaffenreuth are soft and mostly decomposed by weathering, and are generally the richest earths found in the district; then comes the good earth of Kropfmuehle; the others are more hard and solid, require a certain power of disintegration, while the former have unctuous feeling and can be disintegrated by gentle pressure between the hands.

The results of the tests performed in the manner above described, are given in the following table:—

TARLE 41

Graphite Earth from Flakes. Carbon	Carbon.		III	IV	consis	consist of	Carbon
		Ash.	Flakes.	Residue.	Earth.	Carbon as Loss.	Earthy Residue.
% %	%	%	%	%	%	%	%
_	52.68 26.96	11.13	17.44	32.92 44.87	31.82	1.10	83 85 80
	32.84 46.20	13.57	29.24 19.03	50.14 39.25	48.44 30.96	1.70 8.29	$\frac{3.39}{21.14}$
brown earth)	17.35	2.08	23.38	74.69 80.07	56.22 30.9	18.87 49.17	25 28 38.48

From the above table it would seem that a very satisfactory extraction of flake graphite from the ore could be effected, and especially from ore which has undergone a considerable amount of decomposition by weathering, as in the rich earth of Pfaffenreuth. We also note that the tailings under No. 1 carry with them a loss of only 1.1 carbon in the original percentage composition, but that the loss in tailings in 5 and 6 is very high; this may be explained by the fact that these ores contain a certain amount of dense amorphous graphite, which appears to be not in the same degree amenable to the process as the flaky varieties.

In this connection it may be of interest to note that a patent has been issued to Mr. Moritz F. R. Glogner,\* Freiburg, Germany, for the following process in which water and petroleum are used, consisting of the following operations: Purifying the graphite from its heavy admixtures by washing with cold water; mixing the purified graphite with about three or four times its weight of cold water; very strongly agitating the paste within a closed vessel after the addition of a quantity of petroleum of about half the weight of the pure graphite contained in the mixture, and then sprinkling water over the surface of the liquid after the mixture has been allowed to stand in order to obtain a quicker and more complete separation of the graphite particles from the earthy substances.

W. Luzit purifies graphite as follows: The ore is moistened with concentrated nitric acid and then heated. During heating the mass swells considerably in developing peculiar wormlike These forms are chemically unaltered graphite, but in consequence of their fine, delicate structure they are extraordinarily light in weight, so that by washing in water they remain affoat and are carried away, while the constituents of the gangue, which have been completely separated from the graphite by the swelling up of the latter, sink to the bottom by continued agitation of the It appears that graphite, according to this method, can be purified quickly and cheaply; because first, the graphite is not powdered before being moistened with nitric acid, nor is it mechanically cleaned; and secondly, the mass is heated immediately after and further, the swelling is instantaneous. moistening: washing process afterwards takes little time, while little of the nitric acid is lost, as the process is performed in closed retorts.

† Donath, Ibid 89

<sup>\*</sup> Eng & Min. Journal, 1903, page 320

is noteworthy that the graphite obtained by this method is plastic, in a very high degree, so that it can be pressed with ease into plates etc.

The method of Langbein\* consists of the gradual treatment of graphite with sulphuric acid and alkalies, and results in a complete elimination of impurities, with the conversion of the latter into commercial products. Fine pulverized graphite is stirred with water to a paste and sulphuric acid is added. The latter decomposes the silicates e.g. of aluminum under formation of sulphate of alumina. If concentrated acid is alone employed, an incomplete reaction takes place, because the resulting sulphate is insoluble in the acid. The graphite is then separated from the liquid by decanting, then washed with water, until the liquid shows no more acid reaction. The filtrate contains sulphate of alumina, and is treated for the latter. The retained graphite is heated with concentrated caustic soda. The silicic acid, which has been converted into an easily soluble form by decomposition with sulphuric acid, and also the free silicic acid and other impurities are brought into solution. The process leads, therefore, to the production of pure graphite, sulphate of alumina and water glass. If the graphite ore contains silicates like mica, which are difficult to bing into solution, the methods of Langbein can be altered in the follow-

The powdered graphite is mixed with a culculated quantity of ammonium fluoride, then treated with sulphuric acid in the manner above described.

The technical execution of these refining methods, especially when large quantities are to be treated, meets with some difficulties. If the wet method (with acids or bases) is used, vessels made of metal (iron or lead) may be employed, but it must be taken into consideration, that in this case a galvanic element, graphite acid or base metal is created, in which case the metal acts as solution electrode and therefore will be considerably attacked.

The treatment of the carbon with reagents under direct heat, is even more difficult. The best way according to Donath, is to dissolve the necessary quantity of the reagent, caustic soda, sodium fluoride, etc., in water sufficient to form a thick paste with the graphite powder, which is then placed in the crucible. The best crucibles for this purpose are those made of pure carbon, which in order to protect them against burning are inserted in steel crucibles.

<sup>\*</sup> Zeitschrift für angewandte Chemie, Jahrgang, 1900, 354.

The boiling of the graphite with diluted acids and water can be conveniently performed in tanks made of birch or pine wood. It must be mentioned, however, that the finely divided graphite retains stubbornly salts and acids, and therefore it will be necessary to employ large quantities for final washing.

If the graphite contains carbonate of calcium, two methods may be employed: one consists of the digestion of graphite with hydrochloric acid under evolution of carbonic acid gas, and the other by converting the carbonate of calcium into calcium bisulfide, under the influence of sulphur dioxide.

As to the first method, it must be said that not only is carbonate of calcium completely removed if the graphite is finely pulverized, by the action of the acid, but also considerable quantities of other foreign mineral matter like iron, alumina, magnesia, silica and manganese. A Buckingham graphite which contained after drying at 100°C, 13.152% ash, consisting mostly of iron, alumina, magnesia, silica and traces of manganese and a little lime, after treatment with hydrochloric acid contained only 6.69% ash, 6.46% having been removed. this 6.69%, 5.35% consisted of silica, the balance of 1.34% of the other ingredients. From this sample it is apparent that the quantity of acid required for the treatment of graphite, containing carbonate of calcium, is not only determined by the quantity of the latter, but also to some extent by the constitution of the balance of the ash and the quantity of mineral matter therein. Before this process is applied, it is therefore necessary that an average sample of each lot of the ore to be treated be analysed, and the quantity of acid determined and regulated by the results thus obtained. This method can be conveniently carried out on a large scale in circular vats, made of pine, having a stirrer for the agitation of the mass; the pulverized graphite is mixed thoroughly with water and then the acid is added, the whole mass being kept in agitation throughout the process. The latter is finished, when a sample taken from the vats, and thoroughly washed, does not produce any carbonic acid gas by the addition of more acid.

In the second method the finally pulverized graphite is mixed abundantly with water and the whole subjected to the action of sulphur dioxide fumes. The following experiment was made by the writer with graphite from the Black Donald mine, Renfrew County, Ont.: The graphite contained 71.46%

carbon and 8.48% calcite. 10 grams of the finely pulverized ore were placed in a flask, 125 cubic centimeters of water added, and the whole heated and kept at a temperature of 60° C. The fumes of sulphur dioxide (produced by roasting iron pyrites) were passed through the mixture for half an hour. The solution filtered from the graphite was then heated to boiling point, when calcium sulphite was precipitated in the form of white nodules. The graphite was thoroughly washed, and found to contain 79.38% carbon and 1.8% carbonate of calcium. The chemical reaction in this process takes place according to the following equation:—

$$CaCO_3 + SO_2 = CaSO_3 + CO_2$$

This process was carried out by the writer on a larger scale on graphite ores containing from 6% to 14% of carbonate of calcium. The tanks used were rectangular in shape, 12 ft. long, 4 ft. deep, and 4 ft. wide, provided with revolving stirrers parallel to the longer axis of the receptacles. They were filled up to three-fourths of their volume with water, then 750 lbs. of finely pulverized graphite were added, and the temperature of the mass was kept during the whole process at 60° C. The sulphuric dioxide fumes were injected through two inch lead pipes placed on both sides and in the middle of the bottom of the tanks and having a large number of self-acting spring valves. The sulphur fumes were produced by simply roasting iron pyrites in a small brick furnace, from which they were drawn by a pump made of babbit metal and pressed through the pipes and valves into the graphite mass. The treatment lasted from 9 to 12 hours, that is, until a sample drawn evolved no more carbonic acid gas when brought into contact with hydrochloric acid. graphite with the solution was then pumped into a filter press, and when the solid cakes had formed, a stream of water was pressed through the mass in order to clear the graphite completely from the adhering solution.

The whole treatment requires a great deal of care and attention; its success implies that the graphite in the tanks is in an exceedingly fine state of division, otherwise the action of the fumes is weak, and all the carbonate of calcium cannot be brought into solution. The pumps must be made of the proper metal, otherwise they will wear out very fast and the leakage thus created may cause any amount of annoyance and deceptions.

The calcium sulphite produced in this process is used as a disinfectant in distilleries and breweries.

#### CHAPTER IX.

### USES OF GRAPHITE.

## 1.—Refractory articles:—

The properties of graphite specially fit it for the manufacture of refractory articles, such as crucibles, retorts, dippers, stirrers, stoppers, nozzles, etc., and it is principally this industry which gave the impulse to such extensive graphite mining as we have witnessed for the last 25 years.

#### A-CRUCIBLES.

Most of the crystalline variety is used for the manufacture of crucibles, the application of which to iron and steel smelting has resulted in the opening up and development of the graphite resources all over the world. An idea of the increase of the use of this crystalline graphite can be gained from the fact that in the year 1878 in Ceylon, which produces this variety, only 4,230 tons were mined, whereas the output in 1902 amounted to 25,200 tons of a total value of \$3,505,455.

The first crucibles were made in the beginning of the fifteenth century near Passau in the little villages of Obernzell and Hafnerzell; they were used principally in laboratories and in mints for melting the precious metals, and Ferranto Temperato in 1599 is the first who mentions the manufacture of crucibles of flaky graphite. Already Agricola (1495-1550) praised graphite crucibles for their great refractory qualities, and it is reported that they were used, although in small dimensions, by the Alchymists in their endeavour to find the "stone of the wise." crucibles, as manufactured by the "Vereinigte Schmelztiegelwerke." the oldest crucible factory in the world, hold still a dominant position in the industry, although in the course of the last 30 years crucible factories have sprung up in all parts of the world. where iron and steel is manufactured. These Passau crucibles were sold for a long time on the United States market, until the Joseph Dixon Crucible Co'y entered the field; this company began manufacturing in 1827, when Joseph Dixon, the pioneer of this large concern, after careful research and experiments, established the first factory. At the Vienna Exposition, in 1873, Dixon's crucibles, some of them of very large size, were exhibited, and in 1878 an article appeared in the "Jahresbuch fuer chemische Technologie," which contained the following:--" While formerly the only source for crucibles was the village of Obernzell near Passau in Bavaria, America takes to-day an important part in the manufacture of this article, and exports graphite crucibles to all parts of the world, even to Obernzell, which are said to be not only of superior quality, but also cheaper than those made on the spot. The graphite mines are situated in the vicinity of Ticonderoga, N.Y., the crucible factory in Jersey City, both works belonging to the Joseph Dixon Crucible Company. Only the fine, flaky graphite is used for the manufacture of these crucibles, and is ground for this purpose in specially constructed ball mills."

Owing to the tremendous expansion of iron and steel manufacture, a large number of crucible factories were established since the year 1870, both in the United States and Europe, but the large iron and steel works to-day make crucibles themselves for their own use.

In the production of graphite crucibles several factors have to be considered, as difficult combustibility, density, stability and resistance against all mechanical influences. All these properties have to be considered jointly in the majority of cases, and form the primary basis for the manufacture of really good crucibles. At first sight the making of crucibles appears to be very simple, but so many points have to be taken into consideration, especially the selection of the material, that we may say: to make a crucible of distinct refractory qualities is an art.

According to Jochum\* the resistance of a crucible is a function of the chemical and physical properties. In the making of crucibles it is an essential point that the material to be used, that is the mixture, as far as condition and percentage or composition is concerned, is pyrometrically useful, that is sufficiently incombustible. It is necessary, therefore, that the manufacturer must know his material in a pyrometric sense, and in order to make improvements and eradicate the faults, it is specially important to know the exact chemical composition. In a well-guided crucible factory the control of the different phases of

<sup>\*</sup> Paper read before the "Verein Deutscher Ingenieure," 1894.

manufacture by permanent pyrometric determinations as well as chemical analyses of any incoming new material is indispensable. Experience has demonstrated that wherever there is neglect in the exercise of these frequent investigations, the crucibles so manufactured all possess faults of a manifold nature, of which cracking and reduction in the resistance against fire are the principal ones.

Further essential factors, are the physical and mechanical considerations. It is highly important that the various materials to be used are combined together in such a way that the finished article has sufficient density and solidity, but at the same time a certain toughness both under ordinary atmospheric conditions and when exposed to the fire. The size of the grain of the mixtures, their cohesion and homogenity, are considerations which bear upon the manufacture of a crucible of superior quality.

One of the most important qualities of graphite crucibles is their heat conduction. In all true crucibles or in closed fusion the heat units necessary to melt the metal contained must pass through the wall of the crucible. The difference between the general average temperature developed in the furnace outside of the crucible and of that developed inside is quite considerable. This difference is due to the loss of heat in transmission through the wall of the crucible. Hence, the crucible mixture which is the best conductor of heat is the most economical both of time and fuel. In respect to its heat conduction quality the graphite crucible stands far superior to all other kinds. Graphite alone is a ready conductor of heat. It is due to this quality that the charge of a steel melting crucible in the United States is from 95 to 115 pounds; while the charge of the English clay steel melting crucible is seldom more than 60 pounds. Also it is due mainly to this property that the number of charges taken from a steel melting furnace in 24 hours, when a graphite crucible is used, is as high as seven or eight, while in furnaces using the clay crucible it rarely happens that over four charges are taken from the furnace in the same time.

The conductivity of a crucible varies directly with the percentage of graphite used in the mixture. The loss of heat in transmission varies also with the thickness of the wall of the crucible. The higher the percentage of graphite used the more tender is the wall of the crucible—assuming that the parts of the mixture other than graphite are properly tempered. Graphite

adds nothing to the strength of the crucible, but does add to its life and heat conduction—in fact, supplies the latter quality. It is on these principles that the crucible maker must work to determine the mixture of maximum efficiency for any given class of work. No attempt has ever been made to express in figures a value of this heat conduction property.

The principal ingredients of a graphite crucible mixture are clay, fine sand and graphite. Even the best and most refractory fire clay can offer only a small resistance for a short time against molten metal, especially molten steel. But if graphite is added to the clay, the mass resists as long as the graphite is present in sufficient quantities on the condition that the walls of the crucible can stand mechanical pressure and are not damaged in any other way. The graphite plays a very important part in several respects. It prevents even the slightest oxidation of the metals. which as oxide, but not as metal, enter into combination with the constituent of the crucibles; further, the carbon of the graphite increases (as long as it is not subjected to combustion) the infusibility of the clay mass as the latter belongs itself to the very difficultly fusible materials. For this reason ordinary clay crucibles gain in their resistance against fire by "carbonization," that is they are saturated with coal tar, then gently heated in a furnace, where the carbon is liberated and fills the pores of the crucible. The more imcombustible the state of the carbon, the higher is the protection of the crucible against fire. For this reason graphite is employed, which has a very high degree of incombustibility as compared with the commoner forms of carbon. Another advantage in the employment of graphite is its higher conductivity of heat; the crucible can stand sudden changes of temperature without being affected in any way, while the metal therein melts much quicker than in ordinary clay crucibles. Graphite also makes the walls of the crucibles very slippery, thus facilitating considerably the pouring out of the molten metal. The more a graphite combines all these qualities the better is the quality of the crucibles, and for this reason the selection of the one or the other quality is a matter of the utmost importance.

Mr. W. F. Downs,\* E.M., General Manager of the Federal Graphite Company of New York, speaks of the special qualities added to a crucible by the application of graphite in the following manner:—

<sup>\*</sup> Iron Age, May 24, 1900, p. 5.

"Graphite adds another special quality to a crucible mixture, viz., the ability to stand sudden and severe changes of temperature. On this quality rests mainly the life of the crucible. to stand the strain of passing suddenly from a temperature of 2,000 degrees C, or thereabouts to 100 C, and then of reversing this, and of doing it repeatedly, is borne by no other crucible material. I have heated a small crucible to about 1,400 C, and then suddenly plunged it into water, returned it to the fire and repeated it again and again until the crucible had been shocked 20 times. If rung by striking it with the finger or a small bar, the crucible showed no change in the note of its sound until after the twelfth shock: by the time the twentieth was reached the note was nearly gone. In actual service, where the shock to which the crucible is subjected is not as sudden or severe as that just stated, a crucible has been known to stand from 80 to over 100 charges, though the average life is from 20 to 60 charges when used in melting copper alloys or equivalent metals.

"Just how graphite adds these two peculiar properties to a mixture of clay, sand, etc., is not known. If the graphite in a crucible be oxidized completely from it, the crucible will still keep its shape, though very porous; in fact more than one-half of the composition in bulk will be gone. In this condition it is not fit to hold molten metal, though it still will stand a high degree of heat; it very soon fails and develops cracks on sudden cooling. life of the crucible depends not alone on its ability to withstand sudden changes of temperature, nor on its refractory quality, but largely on the reactions which take place in the crucible. In considering the reaction it is desired to have occur in the crucible, it is well to remember that there is always a reducing agent, carbon or graphite, present in the wall of the crucible. Crucible steel makers count very carefully on the throw of carbon from the wall of the melting pot at different stages of its life. An interesting case of great reduction in the life of the crucible in easy work occurred in reducing oxide of tin in a graphite crucible. The reduction was to be accomplished by the addition of charcoal. addition being scanty, the tin oxide absorbed carbon as graphite from the wall of the crucible, and other tin oxide as a strong base fluxed the refractory clay and sand of the wall of the crucibe, with the result that the crucible wasted away in a very few charges. The addition of charcoal in excess increased the life greatly."

The only disadvantage of the employment of graphite is the

decrease of cohesion in the mass with the application of the graphite beyond a certain limit. No strict rules can be laid down regarding the proportion of graphite to the clay mixture, and this must be determined in each special case by a series of tests and is also regulated in the majority of cases by the objects in view.

The methods for the manufacture of crucibles, while differing in some minor details, are practically the same everywhere. clay must be of the finest and purest quality, and not too much stress can be laid upon the selection of this article. There are fire clays to be found in every country, and while they may give satisfactory results in the manufacture of ordinary refractory materials, they cannot, however, in the majority of cases be used for crucibles. The best clays so far found are the Stourbridge (English) clay, the Passau and Klingenberg clay of Bavaria. latter especially on account of its great purity and highly refractory qualities is being used not only in most of the crucible works of Austria and Germany, but finds also ready application in the United States and England. This clay has a color ranging from light blue to dark blue, has a very greasy feeling and aspect and is exceedingly plastic. It swells up when brought in contact with water; when subjected to washing the best qualities show a percentage of clay ranging from 84.98 to 88.95, and of sand ranging from 11.00 to 15.15, the latter containing a slight percentage of A chemical analysis of two clays generally used for crucibles gave according to Dr. Bischof, Wiesbaden, (Germany), 6th July, 1903), the following:—

TABLE 42.

	Clay 1.	Clay 2.
Alumina Silica. Magnesia Lime. Oxide of Iron. Kali. Pyrite.	% 31.16 54.16* 0.38 0.40 1.66 0.97 Trace	0'6 30 43 55.76† 0 27 0 34 1 .37 0 .72 Trace
Loss.	100.21	11 42

<sup>\* 31.11</sup> chemically bound, 23.05 sand with  $22.91^{\circ}_{\ell}$  silica,  $0.10^{\circ}_{\ell}$  alumina.

<sup>† 33.81</sup> chemically bound, 21.95 sand with 21 80% silica, 0.12% alumina.

The clay is first dried in kilns, where the temperature is maintained at 120° C, until all moisture is driven off. The perfectly dry material is then ground in small stamp or edge stone mills and subsequently screened to take out all the lumps or foreign matter which may have been mixed up with the same in handling. If scaly graphite is employed it is important that all the lamellæ have a fine leave like structure without any lumps. Flakes of large size are not desired as they decrease the coherence and density of the crucible. Amorphous graphite is very often used by steel works, which manufacture crucibles for their own use, but for reasons explained in the chapter on the qualities of the graphites it is not advantageously employed for this purpose.

Quartz or pure sand is sometimes added in limited quantities to diminish shrinkage of the mass; also sometimes ground bricks (Chamotte) to prevent bursting of the crucibles and the formation of cracks.

The mixture is then slightly sprinkled with water and left to itself for several days for a thorough impregnation with the moisture; at the end of this period the mass is thoroughly mixed and worked over in a clay cutter; detached quantities are cut out and worked into balls or ellipsoid like forms, which are stored away for several weeks in a cellar or some such receptacle. When these small lumps commence to show a dry crust on the surface, they are again cut up and worked over in a cutting apparatus, until the whole is a homogeneous mass.

The proportions of the various materials, which enter into a good crucible mixture, are as a rule guarded by the various factories as a secret, and as the constituents are of diverse qualities it is obvious that one formula is not applicable for all purposes. So, we have in the case of the employment of Klingenberg clay of a quality as described above and Ceylon graphite the following formula:—

75 parts in weight Klingenberg clay.

25 parts in quartz.

100 parts in Ceylon graphite.

Very often, where high refractory qualities are demanded, the quantity of graphite used lies between 80 and 100, but many factories use far less than this quantity. According to Ledebur,\* crucibles for the manufacture of tool-steel in the larger steel works of Lower Austria, contain only from 33% to 60% graphite. But

<sup>\*</sup> Thonindustrie Zeitung, 1895, No. 3.

the graphite used in these crucibles is the very best Ceylon especially cleaned for the purpose. Very often it is chemically refined by boiling it with sulphuric and nitric acid (3–1 parts), whereby considerable swelling of the graphite is caused. After washing, it forms a loose powder of a specific gravity of 2.25, which does not readily mix with water. The quartz employed must be composed of small grains and should be of the purest quality. If a crucible of an extra high incombustibility is wanted, a fire clay of high pyrometric qualities, specially prepared for the purpose, is added.

The shape of the crucibles is obtained by placing or pressing the mixture into a vessel of the form the crucible shall ultimately possess; these forms are generally made of wood or iron and are sometimes composed of two detachable symmetric parts. The walls of the crucible are formed by pressing the material against the sides by inserting a cone of wood or iron of corresponding size into the mass. This method is successfully used in the large brass works of Achenrain in Tyrol. The forms and tools used in this factory are illustrated in Fig. 35.

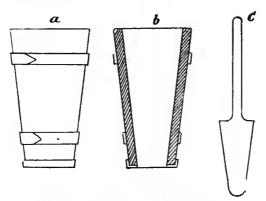


Fig. 35.—Tools for making Crucibles.

The forms (a) and (b) consist of two corresponding halves, (c) represents the handled cone, by means of which the walls and the bottom of the crucibles are formed.

Care must be taken that no air bubbles remain in the clay, which can be effected only by repeated working over and pressing of the mass against the sides of the form. Any material forced out of the vessel by the insertion of the cone is cut off by a wire and placed back into the cutting and mixing apparatus. When

the walls and bottom have thus been made roughly, the cone is taken out, washed, again inserted in a moistened condition, and pressed against the walls with a turning motion, thus causing the disappearance of all uneveness on the inner surface of the crucible. The crucible remains in the form for a day or two, is then taken out and placed on a piece of board, previously covered with powdered graphite. It is exposed to the open air, but not directly to the sun rays, as they cause cracking; the crucible is repeatedly turned on that board to prevent sticking. They must also be inspected frequently to see whether cracks and air bubbles are forming. The latter if found are cut open with a small knife and then pressed gently to the walls with the fingers. Cracks are also closed in a similar way. This continues until the crucibles are perfectly dry and solid.

In the manufacture of crucibles by hand, very often a revolving table is used, upon which the crucible mass is worked over and formed by hand into the desired shape, but this method has the disadvantage that the mass, in order to be effectively worked through, must contain more moisture than when forms are use. Further, the uniformity in thickness of the walls and of the bottom depends entirely upon the practice ability of the manipulator. On the other hand the making of crucibles on the revolving table has the advantage over those made on a stationary table in forms, that the drying in the form is done away with, consequently the latter can be entirely dispensed with, which in large factories means a considerable saving of capital. For small crucibles, however, the use of forms is indispensable, for the renson that a bottom of only a very small diameter would not be strong enough to support the weight of the walls, and consequently the crucible would fall to pieces.

The making of crucibles on a revolving table is briefly carried out as follows:—A lump, sufficient in size to make a crucible, is placed in the centre of the table; the latter is then made to revolve either by the feet of the manipulator, as is still the practice in some of the older crucible works in Europe, or by mechanical means. The revolving lump is worked with both hands with varying pressure and in different directions, from the base upwards and from the core to the outside, whereby a gradual shaping of the mass into the desired form is effected. The thickness of the walls depends upon the size of the crucible, the latter upon its purpose and also to some extent upon the construction of the furnace in which the same is to be used.



Making of Crucibles in Dixon's Crucible Factory, New York.

Making of Crucibles by Machinery:—In some of the modern factories crucibles are made by machinery and there is such a variation in the pieces of apparatus employed, nearly every factory having its own design, that space does not permit the description of a number of them. In some, small revolving plates take the place of the revolving table. The form of the crucible is fastened to the centre of this horizontal plate and receives the crucible mass. The whole is then made to revolve. A cone, which is suspended by a rod over the centre of the form, is then lowered by means of screws into the revolving mass, and by pressing against the sides, the walls and finally the bottom of the crucible are produced.

Another method consists in fastening the form containing the mass in a reversed position in the centre of a revolving table, the cone for the production of the walls entering through an aperture from below.

Picard and Bergman's apparatus consists of a cast iron form, lined with copper-sheeting, into which the crucible mass is placed; a cone made of iron is gradually pressed into the latter, giving thus the desired form, and at the same time the walls the desired density. The rejected stuff leaves the form through small canals. It is claimed for this innovation that the work is done far more quickly than by the ordinary revolving table.

In the works of the Morgan Crucible Company, of London, the graphite crucibles are made in a special machine invented by Morgan and Hyles.

Another apparatus which has been used in European and lately in American factories is described as follows:--In the accompanying illustration Fig. 36, (c) represents the form, (e) the cone, (kk) two long screws with right and reverse thread, (h) a stationary ring plate attached to (kk) in the manner indicated, (b) a plunger moved by hydraulic pressure in (a). The operation of this crucible press is as follows: Form (c) and cone (e) are lowered or lifted by the two screws (kk), set into motion in the manner indicated at their upper ends; in the illustration the position is shown, where the cone is inserted ready to receive the crucible A lump of the latter is placed on top of the plunger (b) which presses the same by hydraulic power into the form (c). After the pressing of the crucible is completed form (c) is lowered and (e) lifted by screws (kk), and the crucible remains on top of (b) held there by the stationary ring (h). After withdrawal of the cone the crucible is carefully taken out from the side.

In some of the American factories the crucible mass is placed in a form made of gypsum; the latter is fastened in the center of

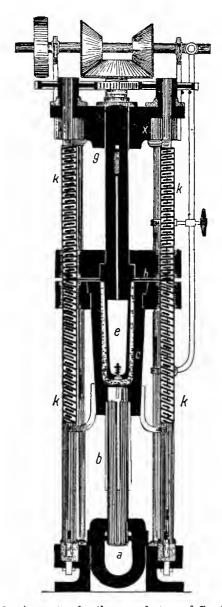


Fig. 36.—Apparatus for the manufacture of Crucibles.

a revolving plate, and the mass is worked into the desired form by centrifugal power in connection with a vertical arm suspended over the apparatus.

Schlickeysen pressed in an apparatus, specially designed for the purpose, 1,500 small graphite crucibles per hour, made of dry graphite powder.

Drying and Burning of Crucibles:—The more slowly the crucibles are allowed to dry, the less liable are they to crack. The drying is generally done in large kilns, the bottom of the crucibles as a rule touching the hottest place. In some factories the crucibles are placed on large drying platforms made of iron, heated moderately underneath, in others they are placed on shelves, the kiln being heated by hot air by a correspondingly large furnace. In the large factory of the Morgan Crucible Company, of London, according to Bischof, the crucibles are dried in kilns, through which the upper part of a large calcining furnace goes, in order to maintain an even temperature.

In the Bavarian factories they are dried for a period of 6, 8 and 10 weeks in large heated kilns, the drying being considered as finished when the crucible on touching with a hammer gives out a metallic sound.

In the majority of cases all crucibles as soon as they are dried, are burned in large furnaces. S. A. Peto\* holds that all graphite crucibles without exception should be burnt before using, otherwise they are liable to burst on account of the dampness absorbed from the air. The same authority says that burning can be done away with by preventing this moisture getting into the crucible. He prepares a mixture of the composition mentioned below, applies the same to the surface of the crucible and exposes the latter to a gentle heat, whereby the vessel adopts a glazed surface. It is claimed that in this way all the pores are filled, thus preventing any moisture getting in, and further that the crucible can be used for melting metals without being burnt previously. A mixture which is said to have given good satisfaction is composed of the following:—

12 parts by weight unburnt clay.

2 parts by weight Cornish clay.

4 parts by weight unburnt clay.

1½ parts ground, red clay with water mixed to a paste.

½ part manganese dioxide.

<sup>\*</sup> Thonindustrie-Zeitung, 1879, 237

These proportions may be altered, but the contraction of the mixture must be the same as the contraction of the crucible. The burning of crucibles is, as a rule, done in large capsules made of very refractory clay, which hold a number of them and are placed in a furnace. The burning makes the crucibles hard and imparts to them a gray white to blue gray color, which, however, must not be taken as a criterion of the quality of the crucible. In Birmingham, according to Bischof, the burning of crucibles is effected in long rectangular wind furnaces of about 30 centimeters square and 65 centimeters deep. The crucibles are placed bottom up on a layer of coke of several inches in thickness: the fire is then started, whereupon the whole furnace is filled with coke, so that the crucible is gradually heated, until it is red hot. The crucible is then reversed and after a second exposure to a red heat, the furnace is left to cool. If the heat is not applied gradually the crucible will crack and burst.

The burning of crucibles, used in the manufacture of steel, is generally performed in two ways, differing from each other only in the kind of fuel to be used in melting the metal: if for instance coke is used in steel making, the burning is done with coke; the same applies to coal. When coke is used the crucibles are placed 16 to 20 pieces at a time in a sort of rectangular furnace, having a large door on one side. The crucibles are placed on a grate several feet from the bottom of the chamber, leaving a space of one inch between them. When the grate is covered with the crucibles, the door is closed, and all the space between the latter is filled with small pieces of coke, and the same ignited. Care must be taken that the fire penetrates the fuel slowly, so that it reaches the surface in about 6 or 8 hours. The longer the exposure to the fire, the better generally are the crucibles: however. the fire must not be allowed to die out in the lower part of the coke, while the centre or surface is still strongly ignited. The reversed position of the crucible has for its purpose the complete burning out of the bottom. If coal is used, as a rule, kilns made of cement and having a door on one side are used. They contain from 15 to 20 crucibles, the latter are placed on a grate either on the side or bottom up. The burning process lasts from 8 to 10 hours.

## PROPERTIES OF GRAPHITE CRUCIBLES, THEIR USE ANDABUSE.

In the early days of the use of plumbago crucibles it was soon found that they would stand very severe service. It was

# PLATE XIX.



Drying of Crucibles in Dixon's Crucible Factory, New York.

even a matter of wonder that any crucible could stand the service and conditions to which they were exposed. Now, however, the manufacturer is often asked to make them, to stand paradoxical conditions. The oft repeated phrase that "graphite is not affected by heat, cold, acid or alkali," and the idea that it is inert under any and all conditions, is largely accountable for this. When we remember that a graphite crucible is composed only partly of graphite, that at high temperature graphite is subject to all the reactions that affect carbon at a lower temperature and that the component parts of the crucible other than graphite are subject to many reactions, it will be readily understood that the graphite crucible manufacturers attempt many things and have opportunity for many failures. They have made many absurd experiments, but have successfully solved many hard problems. If the experimental work had been done systematically and a careful record kept, much better work could have been done. Manufacturers of these goods seldom meet and very rarely exchange views, and much work and experimenting need yet to be done.

Mr. W. J. Downs, late general manager of the Federal Graphite Company, New York, gives his views regarding the use of crucibles as follows:—

"If one studies carefully the conditions to which the graphite crucible is exposed and the nature of the material used in the composition of the crucible mixture, he discovers certain fixed principles, the observance of which will add much to the uniformity and value of the crucible for various purposes. The requisites of a good graphite crucible are refractoriness, strength, heat conduction, long life, i.e., capability of standing many heats—and resistance to the action at high heat of materials in contact within or without. The refractory quality of a crucible is much misunderstood. It is not so difficult to arrange for the refractory quality alone as for the other requisite qualities of a good crucible. A mixture of graphite and high grade fire clay would give a mixture refractory enough to stand the fusion of platinum, but would utterly fail in the other requisites.

"The range of refractoriness required of a graphite crucible is very great. The crucible giving good service at the temperature of nickel fusion is not well adapted for service in spelter casting. This means that the mixture has to be varied according to the temperature of the service required. This would not be

<sup>\*</sup> Iron Age, May 10, 1900, page 10.

the case if the refractory quality were not so intimately associated with the other requisites of a good crucible. The graphite in the wall of the crucible begins to oxidize at a temperature of about 600 degrees C. Its rate of oxidation increases with the temperature and varies with the composition of the furnace gases. life of the crucible depends largely on the non-oxidation of the This is prevented by the production of a "glaze" on the outer surface of the crucible. The production of this "glaze" depends on the refractoriness of the crucible mixture. Hence, if the material be too infusible the life of the crucible is very much shortened. On the other hand, if it be too fusible it softens and fails utterly. The production of the glaze depends on the component parts of the crucible mixture, the temperature of service and the nature of the fuel used. Some users coat the outside of the crucible with a mixture more fusible than the wall of the crucible itself. If the first heat to which a crucible is subjected is high enough to produce this protective glaze its life at lower subsequent heats is much prolonged.

"It is usually more difficult to make a good crucible for low heat service than for high heat work, though this is not so much due to the crucible maker as to the fact that low heat fusions are apt to be overheated. Hence, a crucible should not be too refractory, but should be so made that oxidation of the graphite is prevented at the temperature and condition of service and yet kept strong enough to stand the burden of metal and the handling it is to receive. This last remark has its bearing on the size of the crucible in relation to strength. It is possible for a crucible to fill all the requirements and not be able to stand the burden of metal it is capable of holding, nor the more severe strain of handling by tongs. Every such handling shortens the life of the crucible, and the movement in the direction of the adoption of various types of tilting furnaces for large crucibles shows that the users of crucibles are appreciating this point."

Mr. Erwin S. Sperry\* gives his views regarding the qualities of crucibles as follows: "When a graphite crucible is well made, it has several characteristic features: First, when broken, the fracture has a distinctly fibrous nature, so that instead of being short like a piece of brick or other vitreous substance, it closely resembles a piece of wood. The famous Ceylon graphite imparts this quality to it. Such a fibrous nature is capable of resisting

<sup>\*&</sup>quot;The Brass World," Jan., 1906.

the sudden changes of temperature, and the strain to which a crucible is put in lifting it out of the furnace. Second, a crucible must be well proportioned. The bottom should be thick and the sides gradually lessened in thickness towards the top. When one stops to think of the enormous strain that is put upon a crucible, it will be apparent that the design of a crucible is an important matter. In Fig. 37 is shown a No. 70 graphite crucible which was broken in two. The fibrous nature of the crucible material may be seen and also the varying thickness of the different parts. Crucibles with thin walls possess the advantage that they conduct the heat more rapidly, but their life is shorter, and they are liable to give way under the strain. Experience has shown that the present shape of crucibles is a good one and that the thickness of



Fig. 37.—Half of No. 70 Graphite Crucible.

the walls should vary as indicated in Fig. 37. I believe that the crucible shown in this figure represents a well proportioned crucible.

"The question of the color of crucibles is one of long standing, and only the uninitiated believe that there is any particular virtue in one color. The black and the white crucibles are both of the same mixture, and both black and white ones may be made at will. The reason for this is the fact that the graphite on the

surface of a black crucible has not been burned off in the kiln, while that of the white variety has been removed by the oxidizing effect of the atmosphere. Let us take two crucibles for example, which have just been made and air dried. They will, of course, both be black. Let us assume that one is smaller than the other, so that they may be 'nested,' that is one placed inside the other. In this condition they are placed in the crucible kiln and burned. After the burning operation has been completed, it will be found that the outside crucible is white, while the inside one is black. If the outside of the white crucible is scraped off with a knife it will be found that the whiteness is only skin deep and cannot affect the crucible in any way. The popular notion that the graphite is burned off from the outside to such an extent that the life of the crucible is shortened is nonsense. minute amount of graphite is lost that no appreciable effect can be noticed. A white crucible is as good as a black one and a black one the equal of a white variety."

Graphite crucibles may be classified according to the kind of metal to be fused and the kind of fuel used. These varieties differ in shape and also in composition. The first division is into steel and brass crucibles. Other metals are melted in one or the other of these two varieties. The other division is according to the kind of fuel to be used, and they are known as gas, coke or coal. The reason for these changes is explained in the paragraph on the theory of separate crucibles. The general shape of all these as made in the United States is that of an egg cut off flat at each end. The steel crucibles are of nearly the same diameter at each end or often smaller at the top, and the bilge or greatest diameter is a little more than half way up from the bottom. The brass crucibles are of the same general shape. except that the diameter at the top is considerably larger than at the bottom, the bilge being in about the same position. The ratio of diameter to height is less in steel than in brass crucibles. This ratio is less in all foreign makes than in those made in the United States. The slimmer crucibles are more economical in fuel than the broader American type. Many special shapes are made to suit special work or furnaces or notions, but the egg shape is the prevailing one. The notation of the size of a graphite crucible is quite arbitrary and has changed from time to time. It is based on the holding capacity of the crucible in pounds of metal and is expressed in numbers. At present the unit of size of a brass

crucible is 3 pounds of metal per number, i.e., a No. 60 crucible should nold 180 pounds of metal. As the specific gravity of the metals and alloys melted in these crucibles varies, the holding capacity in pounds also varies. The usual metal meant, however, is ordinary brass having a specific gravity of about 8. Though the size of the steel crucible is also expressed in numbers, the relation between sizes as expressed by the number is more vague. A No. 60 steel crucible is used to melt about 110 pounds, and a No. 50 to melt about 95 pounds. Steel crucibles are usually made only in the two sizes mentioned. Brass crucibles are made in a much greater range of sizes, varying from the tiny jewelers' crucible of less than 1 pound capacity to the 1,000 pound crucible used for melting soft metals. Still larger ones are made, such as the retort shaped crucibles used by the silver refiners in the zinc distillation process. These larger crucibles are not removed from the furnace for casting or charging, the furnace being of the tilting type.

They stand in the manufacture of brass from 35 to 45 heats, where clay crucible would stand only from 4 to 6. The very best crucible, made of the best quality of crystalline graphite, can stand from 50 to 60 and even 70 heats of brass, a crucible made of amorphous or dense graphite stands only from 6 to 8 heats. After every heat attention must be given to the removal of the slag.

In iron and steel melting good graphite crucibles, in which the crystalline variety is used, stand 6 to 8 heats, whereas crucibles made of dense or amorphous graphite stand only one or two heats. Of course it will be readily understood that much depends upon the way crucibles are handled in the foundries, and the occasional disparity of results observed in the use of crucibles is very interesting. According to Mr. Erwin S. Sperry it is of great advantage if the crucible user always assumes that every crucible, which he uses, is soaked with water. By assuming that his crucibles are damp, he will take pains to see that they are carefully stored in a dry place. In using them, he will not subject them to the fierce heat, as is frequently done, but will carefully "anneal" them. On the other hand, if it is assumed that the crucibles are dry and they are immediately placed in a hot fire, it is almost certain that all of them will "scalp."

The crucibles, when they are received from the maker, should be placed in a dry place. If this place is warm, so much the better. In the brass rolling mill trade it is customary to keep the crucibles on top of the muffles. These are warm and dry, and in a short time the crucible is not only dry, but remains so. This practice is universal in the brass rolling mill trade and large quantities of crucibles are purchased ahead and stored on the top of the muffle There is no doubt about the wisdom of this practice, and every brass manufacturer believes in it. Indeed, one manufacturer has been bold enough to make the statement that storing crucibles on the top of his muffles for a year doubles their life. Perhaps this is more or less overdrawn, but there is no question that the policy is a good one, and that the crucibles are increased in life to a great extent. The care with which the brass rolling mill superintendent stores and dries his crucibles is partially responsible for the large number of heats which he obtains on them. It is a noteworthy fact that the greatest number of heats on cruci-



Fig. 38.—Crucible showing Pin-hole from which Metal has leaked.

bles are always obtained in the brass rolling mill trade. As far as the actual welting operation is concerned there is no difference between the brass foundry and the rolling mill, and the mixtures are the same.

After a crucible has been carefully dried, it should be "annealed." By "annealing" is meant, slowly bringing the crucible

up to a red heat. If one desires to obtain the greatest number of heats upon his crucibles and at the same time take no risk in "scalping" them, he should only use new crucibles in a new coal or coke fire. When the fire is started in the morning the furnace is cold, and the draught is poor, and a crucible placed in the fire is gradually raised to a red heat. After the first heat the fire becomes more intense, and while a careful man will be able to use the fire a second time for "annealing" a crucible without taking any risk, the careless melter will be apt to crack or "scalp" his crucible. If a crucible is to be annealed during the middle of the day, the following method should be carried out. After the heat has been removed from the fire, a shovel of coal is put on and the cover pushed off. After the walls of the furnace have cooled down and the body of the fire has likewise been reduced, the crucible is placed in it.

In "annealing" a crucible, it should be placed in the furnace upside down. The wisdom of this practice lies in the fact that the bottom of the crucible is much thicker than the top and by turning it upside down the bottom is away from the greatest heat. After a time the crucible may be reversed and the cover pulled on, so that the heat is gradually raised to a redness. In this condition the metal may be put in, and the furnace coaled up. It is a singular fact that after the first heat a crucible may be abused without assuming much danger in the way of sudden changes of temperature, and the older it grows, the more severely it may be treated. It is the first heat, which needs care and upon which so much depends. Perhaps cracks or fissures are formed by the careless treatment of a crucible in the initial heat, which later may cause pin holes or other difficulties.

A good crucible should wear away evenly, and when it is about to end its "career", slight cracks begin to appear at the top. The crucible has not by any means outlived its usefulness, and frequently a crucible may be used for quite a number of heats after the cracks begin to appear at the top. As a rule, however, only a few heats can be obtained on it. In Fig. 39 is shown a crucible to illustrate its appearance when the cracks begin. In this particular instance the crucible had become quite thin and the cracks at the top had extended so that it was deemed advisable to throw it away. As a rule the cracks at the top of the crucible determine the end of its life.

Mr. W. F. Downs,\* expresses himself on this subject in the following manner:—

"Many users of crucibles add fluxes, as they are called, to purify the metal, or protect the surface. The substances included under this name in the foundries are salt, lime, borax, charcoal and many mixtures bearing special names. The term flux is not generally construed to include the deoxidizing agents, as alumi-



Fig. 39.—Crucible showing the Cracks which begin to form at the top when its Life is nearly ended.

num, the silicates, etc. It is evident from the foregoing that the crucible may be required to withstand the action of either a basic or an acid contact. Luckily, the presence of the large percentage of graphite protects the binding material of the composition for a considerable time. Basic crucibles are usually acid, but differ in degree. Very nearly neutral crucibles are made of first-class quality. It has frequently happened that the user has changed his flux in character or quantity and blamed his crucibles for giving less service. Crucible users, as a rule, are full of little secrets about their practice, and are very chary about accurately describ-

<sup>\*</sup> Ibid, May 24th, 1900, p. 5.

ing their conditions and practice. They would often be surprised if they were aware how many other people knew their secrets. Luckily, for both maker and user this "charishness" is dying out. So long as the crucible metals were made in small quantities in many shops it was difficult to assemble in one place the results of experience, but now that large shops and combinations are in operation the contact of maker and user is much closer. Without



Fig. 40.-No. 200 Crucible which ran 61 Heats in a Tilting Furnace.

the help and intelligent criticism of the user the manufacturers cannot make much substantial progress. Chemistry and metallurgy are being more and more appreciated in the modern shops, and the crucible is bound to receive more attention and criticism. This means that the crucible manufacturer will not be asked to work so much in the dark as to the conditions to which his wares are subjected, and the result will be better quality for all.

The uses of these crucibles may be divided into those of convenience and necessity. The uses of the latter class include fusions of various metals and mixtures for producing homogeneous alloys, the quality of which would be impaired if the fusions were made under exposure to the products of combustion. The former

class includes those cases in which only a small quantity of the material to be fused, or a limited output only, is needed. A few instances will suffice to illustrate both types. A good alloy of copper with zinc or tin or both cannot be made without the use of the crucible, on account of the oxidation or volatilization of both the zinc and tin at the temperature needed to pour the alloy. The same argument which is offered for the use of crucibles in steel making applies to copper alloys, but one seldom hears of an alloy of copper made by other than crucible methods. It frequently occurs that an isolated establishment needs a small casting of iron, brass, bronze, etc. In such cases the crucible serves the purpose, and is a great convenience.

A list of the various kinds of work in which graphite crucibles are used as the melting pot, includes malleable castings, small iron castings, crucible cast steel, all kinds of copper alloys, spelter castings, file tempering, gold and silver melting and refining. Also oblong, square and round shapes are used in liquid brazing, and as calcining trays or boxes for materials requiring careful, even heating without exposure, such as pencil leads, incandescent light carbons, etc. One of the most interesting uses of the graphite crucible is that of a retort. The distillation of metals certainly requires special retorts if the metallic fumes are to be condensed and used. The best instance of the service is shown in the zinc distillation process now in use in all the silver refining works. Here the graphite retorts, or bottles, are used in tilting furnaces and have a holding capacity of 1,500 pounds.

According to Peto it is advantageous to paint the burnt crucibles, when they are still warm (about 100° C.), with a preparation consisting of either resin, or tar dissolved in turpentine or wood alcohol. Great durability and special protection against cracking or bursting is claimed for the employment of this preparation.

As above outlined the principal characteristic of graphite crucibles is the sudden change in temperature they can stand without cracking; they can be used repeatedly, until all the graphite is burnt so that the vessel cannot support the weight of the metal and handling with the crucible tongs. Even if the graphite burns away near the surface, it still remains in the inside of the clay and cannot burn away for sometime on account of the slagging of the clay-mass, which prevents in this way a direct oxidation of the graphite. But in spite of this protection, if the crucible

is exposed to continued high temperature, the mass will be finally entirely deprived of the graphite, and becomes fragile and breaks up. To prevent the premature cracking of the clay-mass, which has thus been partly deprived of the graphite, the crucible is dipped into a solution of a high class fire clay and borax.

Good graphite crucibles are not so porous as clay crucibles, and for this reason do not absorb so much metal as the latter, which is an important item in the case of precious metals.

In the laboratory, graphite crucibles are indispensable, the softness of the mass permits it to be cut, sawn and perforated by drills. When handled with care they are in spite of the softness of the material practically indestructible.

#### VARIOUS GRAPHITE CRUCIBLES.

There are so many compositions used in the manufacture of crucibles serving all special purposes that it would be futile to enter here into a discussion or description of them, so far as they have been made known. But certain standard compositions, as used by factories of world wide reputation, which produce a first-class article, may be given in the following:—

One factory in Bavaria uses Passau China clay, crystalline graphite (a mixture of Passau & Ceylon) and an addition of broken crucibles, previously cleaned. The latter are finely disintegrated, producing the so-called crucible sand and to this is added one third part in weight of Passau China clay and one third part graphite. Another factory uses graphite from Hafnerzell, which contains from 50% to 65% earthy residue, Ceylon graphite, and from one third to one half part of Passau China clay.

The excellent crucibles of the Plumbago Crucible Co. of London are made of Stourbridge clay and 52.6% Ceylon graphite. They consist, therefore, of equal parts of clay and graphite. This Company uses annually several thousand tons of the best crystalline graphite. Apart from the above composition, the graphite is mixed in various proportions with Stourbridge clay according to the purposes in view. The dry mixture is slightly moistened and left to itself for some time for the complete impregnation of the moisture. The mass is then thoroughly worked through in a clay cutter and finally formed into blocks which are stored away for several weeks before being manufactured into crucibles. It is claimed that by repeated storage the mass gains considerably in plasticity.

The crucibles from Hynam, Tanners Hill, Deptford, are reputed to be of such excellent quality as to stand up to 70 heats.

The Birmingham graphite crucibles consist of three parts graphite, two parts Stourbridge clay and one part brick dust, the latter obtained by grinding old bricks and screening through 3 millimeter mesh. Another composition according to Hauston is as follows:—3 parts graphite, 2 parts hard coke, 4 parts of best Stourbridge clay and one part of ground crucible fragments. The whole is screened through a 3 millimeter mesh, then moistened and thoroughly worked through for some time. The crucibles made out of this mixture are burnt gently in a furnace. They are not affected it is claimed, by the greatest heat, serve to advantage for the melting of the hardest metals and do not crack or burst if heated and cooled down in rapid succession. They take from 50 to 60 kilograms of metal and are reported to stand 14 to 16 heats in iron smelting.

A factory in Duisburg, Germany, makes its crucibles, according to Dr. Bischof, of 8 parts clay, 1 part ground brick and four parts of graphite, the latter of the best Ceylon. The crucibles stand 3 heats in steel melting.

The manufacture of crucibles in a factory in Jersey City, N.Y., according to Dr. Bischof is performed in the following way:-The graphite, which is a fine Ceylon, is ground in Cannon ball mills of the description on page 171; it is then mixed with China clay in different proportions according to the purpose for which the crucible is intended. For every 10 parts in weight of graphite seven parts of the well known Klingenberg (Bavaria) clay is taken. A small quantity of ground charcoal is added, in order to impart to the mass a greater porosity. All ingredients are mixed in a dry condition; afterwards water is added and the whole is worked over in a large cast iron cylinder holding 3 tons. Here the mass is thoroughly cut by heavy arms, which are radially arranged round a vertical axis. To each arm four vertical arms are attached, which are flat on the lower end and banded on the point. In a comparatively short time the mass becomes homogeneous throughout, having the consistency of a thick mud, which is then used for the making of crucibles. These are made either by hand or machinery, special designs, however, being always made by hand. The necessary quantity to make one crucible is weighed and placed on the revolving table, where it is worked into the desired form as described on page 234. The manufacture by means of machinery is similar; a form of gypsum receives the crucible mass and is placed in the centre of the revolving table. The operator presses the mass towards the sides, thus gradually creating in connection with the revolving motion, the desired form.

The crucible is left in the form until dry enough, when it is taken out and burnt in the usual manner.

In special cases the quantity of graphite in crucibles must be increased. Thus Tamm\* makes a special crucible lining for the smelting of manganese ores, which require a high temperature and which cause considerable cracking even of the best crucibles, by taking one part of clay and three parts of graphite; he mixes this with a little water into a thick paste and applies the same to the inside walls and bottom of the crucible to a thickness of 15 millimeters. It is claimed that this lining protects completely the crucible against cracking and bursting.

Coke and charcoal are frequently used in addition to graphite in crucibles. In the Royal Foundry at Berlin, crucibles made of 8 parts Stourbridge clay and ground brick, 5 parts powdered coke and 4 parts of graphite gave very satisfactory results. They stood 23 heats of 38 kilos of iron ore each and were exposed to the highest temperatures, without giving any signs of deformation.

Still another composition, t which is used in the manufacture of crucibles for smelting steel and the precious metals, is as follows: 10 parts disintegrated broken chinaware, 10 parts of graphite, 15 parts of asbestos in threads of a length of about 3 millimeters, 3 parts of quartz and 22 parts of fire clay.

The crucibles of the Patent Plumbago Crucible Company, Battersea Works, contain 52.6% carbon, 2.08% hygroscopic water, 45.40% earths; the latter composed of 68% silica, 31%oxide of iron and traces of lime.

According to Mene, English graphite crucibles had the following composition:-

TABLE 43

	I	IJ	III
Silica	51.40	45.10	50.00
Alumina	22.00	16.65	20.00
Oxide of Iron	3.50	0.95	1.50
Graphite	20.00	34.50	25.50
Lime	0.20	0.00	0.50
Water	1.80	2.50	2.00
Loss	1.10	0.30	0.50

<sup>\*</sup> Dinglers Polyt. Journal, 1872, Vol. 106, page 38. † Dinglers Polyt. Journal 206, page 156.

#### OTHER REFRACTORY ARTICLES MADE OF GRAPHITE.

In addition to crucibles, there are a number of other refractory articles in the manufacture of which graphite is used, such as stirrers for mixing and stirring alloys, phosphorizers, refractory bricks, nozzles, etc. A phosphorizer or a phosphorus charger is a device of plumbago crucible material, which is used for the introduction of phosphorus into the molten metal in the manufacture of phosphor bronze.

A sectional and front view of phosphorizers are given in the

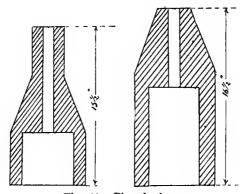


Fig. 41.—Phosphorizers.

accompanying Figure 41. An iron, holding, rod is securely fastened in the little hole on the side of the phosphorizer. The phosphorus, carefully wrapped to prevent spontaneous ignition, is placed in the lower chamber and the phosphorizer is then plunged into the molten metal. The extreme and sudden change of temperature, caused by plunging such receptacles into the molten metal, causes them, when made of ordinary fire clay, to break, and for this reason phosphorizers of graphite crucible material are used; they stand the intense heat without being affected, while the spontaneous ignition of the phosphorous is entirely prevented.

Graphite refractory bricks are greatly in demand for furnace linings and their heat resisting qualities make them unquestionably far superior for this class of application to ordinary fire bricks.

## KRYPTOL.

Kryptol\* is a heat resisting material, made by mixing (in

<sup>\*</sup> Can. Min. Rev , Dec. 1904.

correct proportions) graphite, carborundum and clay, so combined as to form a granular mass. This substance offers to the passage of an electric current a sufficient amount of resistance to generate a high degree of heat without being itself destroyed. It avoids the use of platinum, nickel or any metals which have been heretofore used in resistance furnaces, thereby securing economy and avoiding danger from short circuiting.

The form in which Kryptol is applied, so far, is as follows: An earthenware plate, usually about two feet square, is enclosed in a wooden frame from which, at two opposite sides, project two carbon electrodes which rest upon the plates and which are connected by insulated wires with the current supply. Upon this earthenware plate the granulated kryptol powder connects with both electrodes, a circuit is closed, and the kryptol becomes heated if the thickness is diminished.

Or, by brushing the powder away from one electrode the circuit is broken. If only a thin layer is used, the kryptol becomes heated and will sparkle and glow when the current is on, generating heat so quickly as to raise water to the boiling point within four minutes.

The finer the grains the less active the resistance, and the less heat is obtained; for regulating purposes, therefore, the kryptol is at present manufactured in grains of four sizes. The great ease with which the temperature can be regulated by increasing or decreasing the thickness of the layer on the plate, renders this substance applicable to a great variety of practical purposes. It has been used for heating railway and street cars, houses, rooms, etc.

The new material has been adopted by the laboratories of the University of Berlin, the Technical College of Aix la Chapelle, the Imperial Health Office, and other state institutions in Germany.

#### PENCILS.

### History.

The introduction of the lead pencil may be ranked with the large number of inventions, of which the last three centuries have been so rich, and it can hardly be denied that pencils have played an important part in the diffusion of arts and sciences. Application of lead as a writing material was entirely unknown in the classic age. It was not until the middle ages that its use began. How-

ever this lead was in no way equivalent to the graphite of modern pencils. It was a kind of mixture of metallic lead with some other ingredients, the whole producing a dull, leaden streak on paper. Moreover, at that time lead was used exclusively for ruling and in no way for writing or drawing. It was employed in the form of round, sharp edged discs, similar to those which, it is said, were already used for the same purpose in ancient times. But it is only with the development and growth of modern painting, that traces of pencil like drawings are met with. In the fourteenth century, mention is made by the masters of that time, more especially by the brothers Van Eick, and again in the fifteenth century by Memlink and others, of studies and compositions, which were made by an instrument similar to a lead pencil, upon paper with a chalk-prepared surface. This type of drawing was commonly called "silver-style," a term, however, which was no doubt erroneous, as there could be no question of the use of pure silver in this connection.

In the fourteenth century, drawings were frequently made in Italy with pencils consisting of a mixture cast from lead and tin. Petrarchs "Laura" was painted in this manner by one of his contemporaries, and the method was still in vogue in the days of Michael Angelo. From Italy these pencils subsequently found their way to Germany, but it is not known under what name. In Italy they were called "still," derived from the latin "stylus." However, these articles have never come into general use for drawing purposes. Pens were also used for writing and drawing, and at the zenith of the art period of those days black and red crayons were also in use on a large scale. It is reported that the Italians imported the red crayons from Germany the best black chalk being obtained from Spain.

Vasari writes of a certain sixteenth century artist, that he was equally skilful in handling the stylus or the pen, black chalk and red crayon. It was this period which witnessed the discovery of graphite, a mineral which was soon worked up into an entirely new material for writing and drawing, the "pencil lead." This discovery was made in England during the reign of Queen Elizabeth, for in the year 1564 the celebrated black lead mines of Burrowdale, in Cumberland were discovered. With the opening of this mine the first steps were taken to plant on English soil a lead pencil industry which, in the course of time, was to assume important dimensions.

It is reported that the first lead pencils were manufactured in England in the second half of the sixteenth century. The raw graphite, or "wad" as it was locally termed, was subjected to the following treatment: on reaching the surface it was sawn into strips of the required size, and these, without any further manipulation, were inserted into the wood. Strange as it may appear, the lead pencils first manufactured in this manner are acknowledged to have been the best, and even at the beginning of the present century, they remained unsurpassed upon the score of the softness and fine tone of the lead. Although the Cumberland lead pencils were in great demand, owing to the fact that they were the first to successfully meet the long felt want, they nevertheless owed their permanent and widespread reputation—more especially in artistic circles—to their excellent quality.

The Cumberland pit was kept open for only a few weeks in every year, yet, in spite of this, the value of the graphite raised in this short period, was calculated at £40,000. The mined material was sent direct from the mine to London and put up for auction on the "black lead" market. Sales were made on the first Monday in every month. The average price obtained was from 30 to 40 shillings per pound, but according to Dufrenoy, the best Cumberland plumbago realized as much as £7 per English pound. Of what great importance this mine and the manufacture of pencils connected with it was to England is illustrated by the fact, that the English Government considered it expedient to absolutely forbid the exportation of plumbago in any other form than that of lead pencils. However, though the pit was only kept open for a few weeks in the year, and none of the black lead was allowed to be exported, still the output gradually decreased, until it yielded nothing more than waste material, which was of no value for the manufacture of lead pencils. Seeing these things, it is not to be wondered at that the English looked in every direction for new deposits of plumbago, without, however, meeting with success.

Nothing remained to be done but to find some means for cleansing the impure residue. The method employed for this purpose was as follows: The graphite was taken just as it came from mine and ground into powder, which was then freed as far as possible from foreign substances by chemical action. It was then formed into a compact mass by means of a press, so that it could be cut in the same way as the pure Cumberland lead had

been. In spite of all the art and ingenuity displayed and in the face of all efforts that were made to improve the material, the English have been unable, even up to the present time to obtain any substance, which could in any way replace the natural plumbago as formerly yielded by the Cumberland pit. These conditions operated as an inducement to search for a substitute which would admit of a more economic use of the black lead. Various attempts were made in England to effect this. Attempts were made to combine plumbago with minerals by means of fusion, by mingling it with from 30 to 40 per cent. of sulphur; but sulphur was found to make the graphite too brittle, and the pencils manufactured from this substance gave only almost imperceptible marks. last a combination of antimony was tried; but the substance obtained, although in exterior appearance similar to graphite, gave very unsatisfactory results as a writing medium. Towards the end of the last century the black lead pencil industry was introduced into France, where it soon attained a considerable development. The most important step in the manufacture of pencils is undoubtedly the invention of employing a mixture of graphite and clay, instead of the pure material. This invention was made simultaneously by the French manufacturer Conde in Paris and Hardtmuth in Vienna in the year 1795. The method offered several advantages, for not only did the addition of clay cause a saving of a large percentage of valuable mineral, but it greatly facilitated the method of manufacture, so that lead pencils could be offered at greatly reduced prices.

By these improvements a new era in the manufacture of lead pencils was begun in France. Still there remained much to be done in the field of pencil making in order to do justice to the increasing demands of art and the requirements of more civilized life.

Many different pencils of various degrees were produced, but they did not comply with the different uses for which they were needed. The manipulations of the brittle material required not only deep study, but also conscientious and skilful workmen, in order to impart the necessary standard of perfection to the lead pencil.

In Germany, the industry was only able to develop at a later period and then but slowly, as the transition from the old to the new state of things was a matter of no small difficulty. In this way, many old fashioned forms and methods, which were still clung to with that love created by custom, had first to be removed. Indeed, among the German industries, the manufacture of pencils occupied but a modest place. The first traces of its existence are to be found at Stein, a village not far from Nuremberg. As far back as the year 1726 the church registers mention marriages between "black lead pencil makers" and at a later date, references are found in the same register to "black lead cutters" of both sexes.

But as time proceeded the Bavarian Government directed their attention to this branch of the industry, and did all in their power to encourage it. As early as the year 1766 a Count von Kronsfeld obtained a concession to establish a lead pencil factory in Jettenbach. Later on in the year 1816 the Bavarian Government established a Royal lead pencil factory at Obernzell near Passau and introduced into it the French process, described above, of using clay as a binding medium for graphite. When the manufacture was in full order, the Government transferred the whole establishment to private hands. In addition to this, the manufacture of pencils had also obtained a foothold in Vienna, where a factory had been established, which in accordance with the French method, also mixed graphite with clay.

The great lead pencil factory of A. W. Faber at Stein, so extensive an establishment at the present day, traces its origin back to a very modest beginning: Thus in the year 1760 Kaspar Faber, its founder, settled in Stein and in the year 1761 began to manufacture lead pencils. This was the beginning of the great pencil industry of the district of Nuremberg, which was considered for a long time the headquarters for the whole world for this peculiar manufacture, until a firm from Fuerth near Nuremberg established a pencil factory, at present controlled by the Eagle Pencil Company of the City of New York. This was the beginning of the decline in the large exports to the United States, which will be seen from the fact, that while the export trade in 1890 reached its highest mark having a value of \$169,032.44, in 1893 it fell to \$97,320, since that time it has been declining gradually every year.

To-day, nine-tenths of the pencils used in the United States are of home manufacture, while in addition a very large quantity is exported. The pencil factories of the United States employ to-day upward of two thousand people, paying them about \$700,000 in wages every year and producing about \$2,000,000 worth of pencils.

The City of Nuremberg and surroundings is still the headquarters for the pencil industry of Europe; it has to-day not less than 25 factories and employs about 3,000 persons.

# The Manufacture of Pencils.

The qualities of the different graphites suitable for the manufacture of pencils, have been discussed in a former chapter. The best pencils are made from those qualities, which occur in nature as finely divided scales, which present a material that is more valuable the purer and more homogeneous the variety is.

The literature on the subject of the manufacture of graphite pencils is very limited, in fact there is hardly any publication describing the different methods in a satisfactory manner in the English language; the writer has given in the following a synopsis of all that is known on the subject generally, principally the German manufacture, partly from his own observations during his visits to the largest pencil establishments in Nuremberg, Bavaria, and partly from information furnished in the treatises of Dr. Donath, Professor of Chemical Technology at the Royal Technical Academy, at Bruenn, Austria, and of A. Buchwald (Bleistifte und ihre Herstellung nach beweahrtem verfahren).

Conte used for the production of pencils, a pencil board boiled and saturated with linseed oil, in order to prevent its shrinkage or expansion by the influence of air. The grooves in the pencil boards, which correspond in length and section to the pencils to be made, were filled by means of a fine spatula with the pencil composition, the whole covered with the top piece of the pencil board screwed together and allowed to dry. When the pencil threads are sufficiently dry to detach themselves from the wood, the boards are placed into a gently heated furnace and afterwards are opened and emptied of their contents on a table. If all these manipulations are exercised with care, all pencils will have retained their original straight linear form, while a few or none are broken, which is, of course, a matter of great importance.

In later years, Conte employed instead of wood, which is always liable to be influenced by dampness in the air, brass or copper plates. The grooves for the reception of the lead were close together, and, after drying, the pencils were removed from the metal plates by means of a peculiar instrument invented by Conte. This instrument consisted principally of a number of small iron blades arranged on two cross bars in such a manner that in gliding tha same over the copper plates in the direction of the grooves, the pencils are all removed and placed on a table.

Another method for the making of pencil threads is described as follows: A number of fine iron or steel bars representing the accurate form of the pencils (allowing, however, for a shrinkage), are fastened to the bottom of an iron box in an upright position. Into this box is poured an alloy of metals of a low melting point, like tin, lead, bismuth, etc. After cooling, the iron bars are removed and the whole represents then a block of metal containing numerous tube like holes, which serve for the reception of the graphite mass. Into these forms the latter is pressed; after drying near a furnace, the pencils, loosing in volume through shrinkage, can easily be taken out and further dried in the open air.

Thompson published in the "Records," the following information regarding the manufacture of pencils in England between the years 1810 and 1830. Three kinds of pencils were made: ordinary, ever-pointed, and plumets, which consisted of one-third sulphur and antimony, and two-thirds graphite. The manufacture of the ordinary and ever-pointed pencils commenced with the cutting of cedar wood into short thin strips, which were divided into top and bottom pieces, the latter received in addition grooves of a small square section. Natural crude graphite is then taken and cut into long thin pieces of the same section, fitting thus in grooves of the bottom plates inserted in the latter; any small lumps projecting over the grooves are cut out; the top piece is then glued on, the whole divided into single pencils and the latter turned and finished in machines specially constructed for the purpose. pencils made in this manner, especially the ever-pointed, cost 2s. 6d., and a genuine pencil clad in cedar wood, fetched 6d. cutting of the natural graphite, or of the hard mixture, was performed either by hand or by a fine saw operated by hand or foot. But as the natural variety of such pure quality as demanded for the manufacture of these pencils occurs very rarely, and there is also much of it lost as waste, efforts were made already in the eighteenth century to utilize all this waste material and also instead of the compact graphite, the earthy or scaly natural variety was employed. This, however, could only be done, if the graphite was finely pulverized, sufficiently cleaned from its accompanying foreign matter and then converted into a clayey mass by the addition of a binder or hardening substance. At first, the earthy graphite or the waste, resulting from the application of the compact variety, was formed into a solid mass, and the latter, after hardening, was then cut like the natural graphite in the same manner as described above, with fine saws into fine pieces of small square sections.

Only in later years the method of producing the graphite strips or threads, by pressing the semi-solid mass through fine tubes of the desired section, came into general use.

But the principal difficulty in the manufacture of artificial pencils consisted in obtaining a mass, which in respect to softness and coloring power, was not inferior to the natural compact graphite. Binders, like glue, ising glass, gum trachacanth, and gum arabic, had to be added in such quantities in order to produce a mass of sufficient cohesion that the mass became too hard to leave a satisfactory mark on paper; on the other hand, these binders, in comparison with the unctuous graphite, possessed qualities which detracted from the homogenity of the mass so desirable in good pencils; further, the material under the influence of humidity dissolved, and was then perfectly useless. For this reason the employment of glueing material was abandoned, and colophony (cleaned pine resin), was used instead, as a binder for graphite.

However, it was soon found out that a graphite mass made with the addition of colophony was too brittle, and small quantities of wax and tallow were introduced into the composition. black was also used to some extent, in order to impart to the same a higher coloring power. However, all these compositions proved to be of inferior quality, compared with pencils made of natural block graphite, as it was not until Conte, in the year 1795, revolutionized the manufacture of pencils by the introduction of clay into the composition mass. Graphite mixed with clay in the right proportion is of such plastic qualities, that it can practically be molded with ease into any desired form; after drying, the mass is gently burnt in a furnace. The principal advantage of this process over all others, and also over the natural block graphite, lies in the fact that the pencils cannot only be produced much cheaper. but that the manufacturer, by a greater or less addition of clay. and by burning the mass to a higher or lower degree, can regulate the degree of hardness, while even with the very costly natural block graphite, on account of its greatly varying quality, a pencil of fixed standard quality cannot be produced.

Conte's son-in-law, Humblot, and also Hardtmuth, per-

fected this process of manufacture and introduced gradually modern apparatus and machinery, most of which with slight improvements are still used in all modern factories.

Pencils made with clay as binding medium, compared with those cut out of natural block graphite, have further the great advantage that the lines produced on paper have not such a highly metallic lustre, they appear more distinct and are of a deep, black color. To effect this condition in the manufacture of the finer grades of pencils, a small quantity of lampblack is added, while for ordinary quality powdered coal is used.

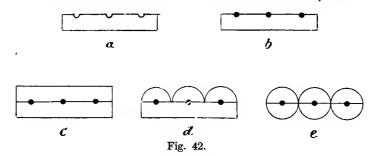
In order to test whether graphite is sufficiently fine enough, a small quantity is mixed with clay, the mass dried and then burned. If the graphite is of the desired fineness, the mass by cutting with a knife must not show any glittering particles, but if the latter are present the pulverization of the graphite must be continued. lampblack or other powdered carbon has been added, the access of air in burning the mass must be carefully avoided, as otherwise the fine carbon particles are liable to be burnt away. The mixing of the materials entering into the composition is performed in a dry state, then water is added, until the mass is a thick paste. In this state it is ground, until a test (as outlined above) does not show any more glittering particles of graphite. By a filter-press this paste is brought down to the consistency of a thick plastic mass, and the latter is then subjected to a series of cuttings, rolling and pressing, until all air bubbles have disappeared, and the material presents a homogeneous mass throughout. The large pencil factories for the production of the graphite clay mass use perfected machinery and a number of specially constructed apparatus.

The pencils are made by pressing the mass, deposited in a cylinder, by means of a plunger through a number of specially constructed mouthpieces, which impart to the pencils the desired thickness and section. The long thread-like pieces issuing from the the apparatus are cut to the desired length and dried, whereby they shrink a little. After drving, which is secured by gentle heating, the pencil lead is burnt in a furnace. The higher the degree of heat and the longer the pencils are burnt, the harder they become, the composition proportions being the same. The degree of heat, also the duration of the same, admissible for the various compositions is to be determined beforehand by a series of tests and by experience. By means of a pyrometer it is not difficult to maintain the proper degree of heat in the furnace, so essential for the hardness of a

specific grade of pencil. The burning or heating of the pencils is performed in air-tight crucibles or fire clay capsules, into which the pencils are placed with a mantle of carbon dust. Low grade pencils are often burnt without the addition of the latter in ordinary Keramic furnaces.

If the temperature during the burning process is raised quickly, the pencils become brittle, they break, burst or bend and they cannot be used again, as the fragments by the burning process have lost many properties of the original composition which are essential for the production of pencils, the most important one being the plastic qualities. Ordinary pencils, which have a tendency to break easily in writing or sharpening, show this fault because in burning them the heat has been raised too quickly. Pencils used for technical drawings or for stenography should conserve their point for a comparatively long time, while at the same time they should exhibit a certain degree of softness. this purpose pencils, which are often used for architectonic drawings are, after burning, submitted to an extra treatment, being saturated after renewed heating with a solution of wax. However, this treatment has only a very limited application, since lines drawn on paper with these pencils cannot be erased by rubber. because the latter glides over the pencil mark without attacking them.

After burning, the pencil threads are placed into the grooves



of the previously prepared pencil board Fig. 42 (a) and (c) and all uneveness on the latter removed; these pencil-boards may contain from 3 or up to 12 or more pencils, according to the quality and structure of the wood employed, the latter for the better class of pencils generally consisting of cedar wood. The top piece, see Fig. 42 C, is then carefully glued on, and the whole, after drying,

cut up first into single pencils of a square section, then into the desired form, round or octagon, polished by specially constructed apparatus and finally stamped and marked, when they are ready for the market.

The manufacture of pencils, according to the foregoing description, is therefore divided into the following operations:—

- 1-Preparation of the mixture.
- 2—Production of pencil thread by pressure apparatus.
- 3—Hardening o the pencils by burning.
- 4—Preparation of the pencil boards for the reception of the pencils.
- 5—Cutting of the pencil boards into single pencils, and cutting and polishing of the latter for the market.

There are, of course, a great many recipes for the composition of pencil lead, practically every manufacturer has his own standard formula, which to a more or less degree is dependent upon the quality of the materials employed.

The quantitative proportions, in which both materials are mixed, vary according to their quality and that of the pencils desired. Two parts in weight of graphite and three parts of clay or even quantities of both are the best proportions for ordinary pencils, but it is evident that numerous grades in hardness and smaller or greater coloring power may be produced. According to Frechette it appears that 4 parts and 8 parts of clay to 5 parts of graphite is the outside limit of the proportions in the mixture. The pencils are the harder and of less metallic lustre, the greater the quantity of the employed clay, the softer, the more shining and of higher coloring power, the greater the quantity of graphite present in the composition mass.

Schuster makes his pencils of 30 parts of graphite, 9 parts of clay, and 9 parts stibnite (gray antimony Sb<sub>2</sub> S<sub>3</sub>) and one part of tallow. The graphite is first ground, and cleansed by washing, then dried and burnt for 2 hours. The tallow is dissolved and then added to the mixture, which is then thoroughly worked through in a lead mill. After burning, the pencils are put into boiling wax. If a greater degree of hardness is desired, clay is substituted for soap. Very hard pencils for drawing purpose are made of the following mixture: 36 parts of graphite, 18 parts clay, 8 parts stibnite and 2 parts lampblack.

Hirnschall, in Vienna, makes pencils out of sulphurized coal and graphite. The latter is cleaned by boiling it for 24 hours with a mixture of water, nitric acid and hydrochloric acid and finally washed with water.

Weilhein, in Vienna, takes 2 parts washed graphite,  $5\frac{1}{2}$  parts clay, the precipitate of  $1\frac{1}{4}$  parts alum by means of dilute caustic potash, and the precipitate of  $2\frac{1}{4}$  parts lead acetate dissolved in 54 parts of water, effected by zinc.

The successful employment of clay for the purpose of pencil manufacturing is based upon its quality to harden when heated, and as the degree of hardness is effected by the temperature applied, it is evident that pencils of different qualities can be manufactured at will. It is necessary, however, that the clay before employment is thoroughly weahed by treating it with clean water and left to rest for about 20 minutes, during which time the sand and all heavy impurities will settle to the bottom. The milky liquid is siphoned off into another vessel, left to itself for sometime, when the pure clay will settle to the bottom while the clear water remains on top to be drawn off. The clay is then filtered and dryed, when it is ready for use. The graphite is ground in a mill of the construction as shown in Fig. 43.

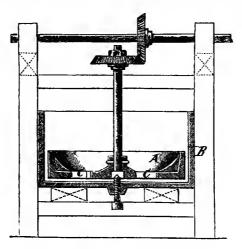


Fig. 43.—Conte's Graphite Mill.

After grinding, it is filtered and dried. The washing of graphite, however, is not always necessary, as in most cases the same is done in the large refineries at the mines. In order to im-

part a certain metallic lustre and softness to the graphite, the latter, after grinding and drying, is placed in a crucible which is covered and then exposed to a white heat.

Conte's graphite mill as illustrated in Fig. 43 is used largely in pencil factories for the mixture of the different materials and is constructed as follows:—

The same is made of cast iron and consists of two pieces, of which the outer one B forms the stationary casing and the inner one A the runner or grinder, set in motion by cog wheels in the

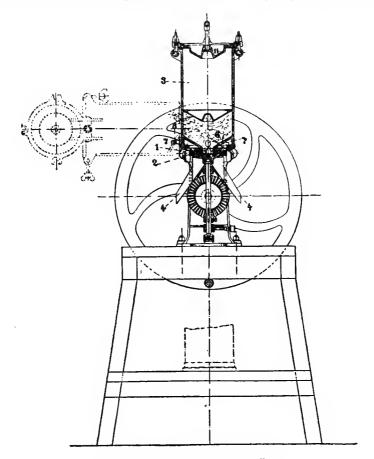


Fig. 44.—Talmie's Graphite Mill.

manner indicated. The funnel shaped vessel A contains 4 holes C in the bottom, which serve as feeders. The apparatus works as

follows: the mass enters through C between the 2 vessels, is ground and by the rapid revolution of A is thrown against the walls of B and up again into the vessel A, thus working the mass over and over again until it is of the desired fineness.

Another apparatus which has given great satisfaction is the cylinder-grinding apparatus system of Talmie, illustrated in Figs. 44 and 45. It consists principally of 2 grinding stones of Swedish granite, (1) and (2) and a cylinder (3) for the reception of the material.

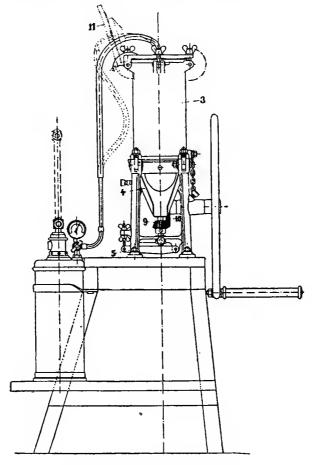


Fig. 45.—Talmie's Graphite Mill.

In the construction of this apparatus the idea has been to make the under stone (2) the runner, while the upper one is made stationary; it is urged in support of this departure that the faces of the two stones will always be parallel by mak ng the spindle, which is attached to the runner perfectly adjustable and automatic. The working principle of this apparatus is very simple: the material is fed into the cylinder, is then pressed by a weight or by air pressure into the space between the stones and is discharged on the periphery of the latter into vessels placed under the mill.

The lower stone or the runner can be adjusted by set screw (5), according to the fineness of the material desired. An endless screw (6) attached to the upper stationary stone supports the feeding of the material to the stones. A hollow chamber (7) is left in the casting, between the material to be ground and the upper stationary stone, for the circulation of cold or warm water or steam, which can be fed through a connection (8). The apparatus is operated from below in the manner indicated either by hand or machinery. For the purpose of convenient cleaning, the cover (11) can be easily removed, while the cylinder (3) and with it the stationary stone 1 can be tilted over in the manner shown by dotted lines in the Fig. 44. By this arrangement the grinding surfaces of the stones can be cleaned with ease. Most of the machines are constructed for forced feeding by air pressure, and for this purpose have a small air compressor with receiver attached to them. contents of the air receiver is sufficiently large for one whole cylinder charge.

The advantages of this apparatus are briefly as follows:—

- 1—Compared with Conte's grinding apparatus described above, which is intermittent, it is continuous in its operation; the material needs to be fed only through the cylinder, and discharges itself after grinding.
- 2—Owing to the great velocity, it can be operated with, it has a large capacity.
  - 3-It is easily cleaned.

The mass coming from these apparatus, having gradually become a thick paste must then be worked by hand similar to a process practised in factories of porcelain ware. It is cut repeatedly by a series of wires, of flat irons, is rolled into fine leaves and lumps and worked thoroughly on granite plates, until all air bubbles and chambers have disappeared and the material forms a homogeneous mass. In this condition it is stored away in a damp place for some time.

For quicker work in larger factories, machinery of various descriptions has been introduced. One of these is illustrated in

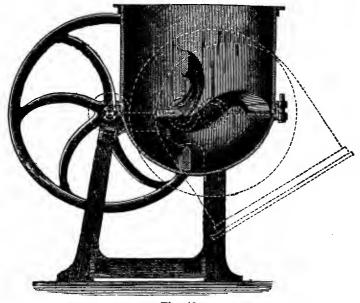
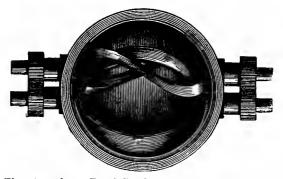


Fig. 46.



Figs. 46 and 47-Pencil Lead Mixing and Cutting Apparatus.

Figs. 46 and 47. This apparatus consists of a vessel, in which two mixers or wings made of wrought iron revolve in opposite directions. As these mixers by their peculiar bended construction, clean each other with each revolution and also scrape off the material from the bottom of the vessel, it is evident that a thorough mixing and working over is effected, so that in a compara-

tively short time, with little power, a completely homogeneous mass is produced. In order to empty the vessel, the latter is tilted over into the position indicated in the Fig. by dotted lines, the working of the mixers, however, being continued in order to clean the apparatus from all adhering material.

As mentioned before, the production of pencil threads is performed in so-called graphite presses, of which there are a number of various constructions in use.

In principle, these presses all consist of a cylinder with a plunger, the size of which differs according to the quantities to be treated. The smallest are four feet high, with a plunger attached to an endless screw of 2 inches in diameter. The head of the screw bears a cog wheel, driven by endless horizontal screws attached to the driving mechanism.

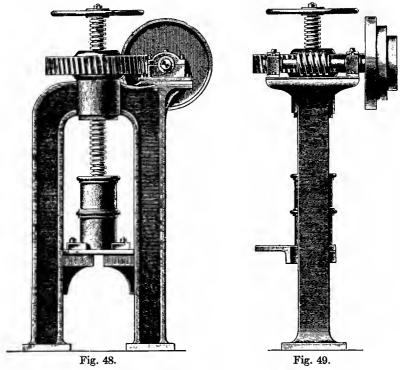
The plunger consists of iron and wood, and fits accurately in the cylinder, the latter contains at the bottom a plate with small holes, corresponding with the size and section of the pencils to be produced. To operate the press, the plunger is removed by an upward movement of the endless screws, the cylinder is filled with the graphite mass, the plunger is again inserted in the cylinder and slowly pressed upon the mass; the latter will then issue through the little holes at the bottom of the cylinder in thread like forms. The threads are allowed to attain a length of from two to three feet according to requirements, and are received on a polished board.

Nearly all modern pencil presses are constructed according to the above description, and the firm L. & C. Hardmuth still employ some of these presses as illustrated in Figs. 48 and 49 in their simplest form.

According to Kohn,\* the pencil presses consist of a vertical cylinder, into which fits a plunger moved by an endless screw. The bottom plate of the cylinder consists, instead of a plate of bronze or steel, of a conical steel ring, in which after having temporarily fastened a small piece of steel, corresponding with the section of the pencil thread to be made, a liquid metal or alloy is poured round; after cooling, the steel form is taken out. The advantage of this mode of manufacture is its easy replacement and repair. The graphite mass is forced through this hole, and the threads produced in this manner are placed on a horizontal polished board. After drying for several hours,

<sup>\*</sup> Karmarsch-Horens, techn. Worterbuch.

they are placed in the grooves of boards and covered with plain boards in order to retain their straight linear form under further drying.



Figs. 48 and 49.—Pencil Lead Press.

According to Buchwald, the new presses are constructed in such a way that by changing he mouthpieces in the bottom plate of the cylinder, different forms and thicknesses can be produced with comparative ease. A very ingenious device is attached to machines of modern construction. The threads, after leaving the cylinder, are received by an endless moving belt, whereby a bending or breaking of the same is very difficult; a cutting apparatus, attached to the machine at a convenient point, cuts them also into the desired lengths. It is of the utmost importance that these threads retain their original forms under all circumstances, as the slightest deformation prevents them from fitting in the grooves of the pencil boards.

It is evident that the latter, form mathematically accurate

canals, and that bent or otherwise de ormed threads, even if they differ only 1-100 of a millimeter from the straight line, cannot be placed in the grooves. Threads which show the slightest deformity, in order to be again used, must be mixed with new composition material, and must go through the same process. In many modern presses the cylinder is heated, so that the threads leave the latter in an almost dry condition, whereby the subsequent drying process is considerably shortened.

In Fig. 50 such a press is illustrated. In this apparatus (ee) are the cylinders for the reception of the graphite mass, attached to a heavy plate (c). The rods (ff) hold on their lower ends the plungers for the cylinder and are fastened at the top to a crossbar (i), which is in connection with a plunger moving in the upper hydraulic cylinder k. The cross-bar (i) is also fastened by two rods (o) to a second plunger (r) at the top of the apparatus, moving in the hydraulic cylinder (l). Hydraulic power is furnished by pumps, which press the water either into the upper cylinder (l) or the lower one (k); it will thus be seen that the plungers on (ff) can be pressed into the cylinders (ee) or lifted at will by water pressure produced in the two cylinders k or (l).

The cylinders (ee) are surrounded by a jacket, in which exhaust steam or hot water circulates. By this arrangement the pencil mass is dried to a certain extent and leaves the bottom plates through the small mouthpieces which correspond with the section of the pencils to be made, in a warm, almost dry condition. The attachment of thermometers to the two cylinders is advantageous, as the graphite mass, when too hot, can easily block the small mouthpieces. In order to cool off the pencil lead coming from the cylinder (ee), a small ventilator is attached to the latter, which continuously throws cool air upon the threads.

The whole apparatus is easily handled by a lever (p); by the latter the plungers can be pressed in the cylinders or their movement reversed or arrested.

The burning is one of the most difficult operations in the manufacture of pencils; much care has to be exercised, as otherwise the latter become refractory and useless, and the material cannot be used over again. If the fire is allowed to rise too quickly, bursting, cracking and bending of the threads will be the consequence and the fragments, which contain now the clay in a burnt condition, have become brittle and are of no use for

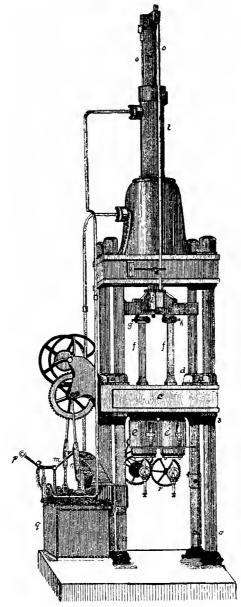


Fig. 50.—Improved Pencil Lead Press.

any purpose. The greater the degree of heat, the harder are the pencils, the composition being equal, and vice versa.

For every mixture, according to whether hard or soft pencils are to be produced, the degree of heat to be applied to the pencils has to be determined beforehand by a number of experiments, generally conducted with the aid of a Wedgewood pyrometer. The burning is performed in crucibles, in which the pencils are placed in an upright position; the spaces between them are filled up with powdered carbon, while a layer of the latter of several inches in thickness covers the whole. The crucible is then tightly covered and placed into the furnace specially constructed for the purpose.

Conte gives the description of a very complicated furnace, which on account of its intricate construction can only be applied to the finest quality of pencils.

Pencils of great softness after burning are dipped in hot wax, whereby they gain a certain amount of solidity.

According to a series of tests made in the earlier years by Conte and Humblot, it has been demonstrated that it is not possible to manufacture pencils of any desired degree of hardness and at the same time of equal uniformity only from graphite and clay.

Conte and Humblot, however, have succeeded in doing this by dipping the dried pencils in a solution of salt of different concentrations and by drying and burning them afterwards. The mass obtains a greater hardness without loss of uniformity. The most useful substances for this purpose are the sulphate salts, e.g., sulphate of soda.

Since the introduction of very efficient wood cutting machinery for the last ten years, especially of American make, the handling of the woodwork in connection with the manufacture of pencils has been entirely revolutionized. While the execution of the different stages in the preparation of the pencil covers was formerly performed by hand or by some other apparatus of primitive design, and was consequently very slow, to-day all this work is done by a series of machines specially constructed for each operation. As to the kinds of wood used, it must be said that for ordinary pencils, pine and fir, and for pencils of better quality, cedar wood of different kinds is employed; of the latter varieties may be mentioned (Juniperus, Virginiana), the white cedar wood, (Cupressus Thyonides), and the South American species (Cedrella Adorata).

In order to give an idea of what quantities of wood are necessary to satisfy the demand for lead pencils, which amounted to nearly 350,000,000 pencils in 1906 in the United States, it may be said that there were required 110,000 tons, or 7,300,000 cubic feet of wood, so that each day in the year 300 tons, or 20,000 cubic feet, of wood are used for pencils. Since practically all the wood is red cedar, and since the pencil industry is steadily growing, the supply of red cedar is greatly depleted; yet no substitute has been found for it. Leaving out the consideration of imported pencils, the average educated American over ten years uses six pencils of home manufacture each year. Ten years ago he used less than five.

Red cedar has a soft straight grain, and when grown under best conditions is very free from defects. Because of its peculiar qualities no equally good substitute for it has ever been found, and it is doubtful if any other wood-using industry is so dependent upon red cedar. In fact, red cedar suitable for pencil manufacture is the only wood, the price of which is quoted by the pound.

Strange as it may seem, no steps have heretofore been taken to provide for a future supply of red cedar. This has been largely due to a lack of information on the rate of growth and the habits of the tree, and to the widespread belief that second-growth red cedar never reaches merchantable size.

In accordance with its policy toward the conversation and economic use of commercial woods the United States Government Forest Service has made a careful study of red cedar and has come to the conclusion that it can profitably be grown in regions of its development. Several changes are recommended in present forest management in order to secure the desired growth. In the Southern forests the cedar will have to be given a better chance instead of being considered as now, a negligible quantity in its younger stages, and many of the forest-grown trees which are now cut for fence posts can profitably be left to attain their full development and thus become available for pencil wood.

The first essential of the wood to be employed is its complete dryness and indifference to atmospheric conditions; without this the covers will warp and cause a breaking of the enclosed pencils; it must also be of straight linear grain and of a certain softness, primarily for purposes of easier handling in their manufacture.



Lead Presses in the Pencil Factory of A. W Faber, Stein, near Nuremberg.

The newer method is apparently more complicated, but true to the principle of the division of labor, is far more satisfactory and effective than the older methods by hand.

As mentioned in a former chapter, wooden slabs of two different thicknesses are produced, one for the cutting of the grooves for the reception of the pencil lead, being the thicker one, and the other serving as the cover.

The grooves are made by an apparatus of the construction as illustrated in Fig. 51—(a) is the working table, to which is

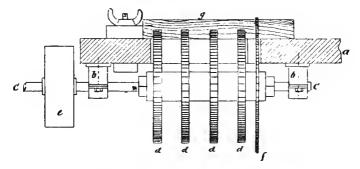
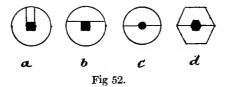


Fig. 51.—Apparatus for growing and cutting Pencil Boards.

attached by means of hangers (bb) a shaft (c), driven by pully (e); dd are the circular saws for the cutting of the grooves, the teeth of which are made either square or half round or three sided, according to the sections desired. (f) is another fine circular saw, cutting off the pencil board into slabs containing 4 pencils each. The working of the apparatus is simple: the slab (g) is moved against the revolving saws (ddddf), whereby the grooves are cut and at the same time the pencil boards are cut to the desired width.

The final stage of manufacture comprises the insertion of the pencil lead, the covering and cutting into single pencils, polishing, printing and packing of the latter for the market. The pencil boards with the grooves up are placed on a table and covered with a coat of glue. The pencil lead is then placed into the grooves, which may be of two kinds. One kind has the same section as that of the pencil thread, and the latter fits accurately in the same Fig. 52C, the other kind is large enough to leave a little space over the pencil lead, to be filled later on with a strip of wood, Fig. 52a. Or if the pencils and threads



are of circular or six sided section the grooves of each pencil board are of the same section, and the latter represents therefore equal halfs, see Fig. 52c and (d).

The other half of the pencil board or the cover is then glued to the one containing the pencil lead and the whole securely and evenly pressed in common screw presses, until they are dry. They are then taken out, cut into single square pencils, and the latter subjected to further cutting and planing in special machines, until they have the desired section, round or six sided. Most of the pencils, especially those of a better quality in order to improve their appearance, go through polishing machines and through printing presses, to have the trade mark or the name of the manufacturer put on.

#### OTHER USES.

Apart from the manufacture of refractory materials and pencils the real value of graphite is yet only beginning to be understood and its possible uses in the future can hardly be forecast. The application of the mineral to certain manufactures has created quite a large industry in itself, and while it is not the purpose of this treatise to enter into a discussion of the different manufactures in which graphite plays a prominent part, still it may be of interest to outline the principles upon which the value of these various products are based.

## GRAPHITE AS A LUBRICANT.

With the introduction of heavier machinery the service demanded of a lubricant has become more and more severe. For much of this work it is found that oil will not answer at all, and for much more it answers only at great expense; hence, the use of greases and the more solid lubricants, such as graphite, mica, soapstone, sulphur, etc. A great deal of study has been given to the subject of lubrication; much has been written and printed about it, but it is all simmered down to one proposition, which is

given in the following words of a prominent engineer; "The more solid the lubricant that can be used, the better the lubrication."

When graphite first began to be used as a lubricant anything, which gave a stove polish lustre when rubbed, was assumed to be "black lead" and fit for lubricating purposes. Experience soon proved it to give very varied results—sometimes very good and sometimes the reverse; in fact it was not reliable, because of a lack of uniform, correct sizing and purity, and soon fell into disrepute amongst practical men though it continued to be well spoken of in books. In 1868, however, systematic experiments were begun in the United States with a view of producing a reliable lubricant from graphite and the final result has been very satisfactory.

Lubricants are divided into three classes:—Fluid, or oil lubricants; pasty, or grease lubricants; and solid or dry lubricants. Of the substances used in the third class the most remarkable is graphite, the value of which as a lubricant is so generally conceded, that it seems strange that its use is not more extensive. All users of machinery know its value and use it in an emergency as a "cooler" of hot journals. While this use is general, the amount used is not at all commensurate with its merits.

Graphite has a very low coefficient of friction. It is composed of but one element, carbon, hence is not affected by the action of acids, gases, alkalies, or by considerable changes of temperature. It oxidizes slowly at a temperature above a red heat. When subjected to the action of any of the conditions mentioned. it retains constantly its peculiar property of low coefficient of friction. Graphite is soft, and adheres readily to metallic surfaces when subjected to light pressure. This property causes it to fill the pores and to even up the roughness of metallic surfaces in rubbing contact. The surfaces so coated are thus covered with a veneer of allotropic carbon or graphite, which reduces the coefficient of friction of the rubbing surfaces to practically that of graphite itself, and also protects those surfaces from the action of vapors. This applies particularly to cylinders; hence the special value of the use of graphite in cylinder lubrication, where high pressure steam, oil, or gas is used.

For many years the accepted type of a lubricant was an oil or fat, but these bodies, whether of vegetable or mineral origin, are liable to become rancid and break up into other compounds under the action of the heat and vapor to which they are exposed, and often exert a corrosive action on the metal surfaces.

Soon after the discovery of petroleum in 1859 mineral lubricating oil came into use. These hydrocarbon oils were free from the danger of corrosive action, but lacked body or viscosity under heat or severe service. This was followed by the compounding of the mineral oils by the addition of the body-giving animal oils. These compounded oils do not become rancid, as the animal oil is sealed from contact with the air. For many years these compound oils have met the requirements of cylinder lubrication.

The use of high pressure steam, oil and gas in specially constructed engines has necessitated a much higher temperature in the cylinders. All the oil lubricants lose much of their body or lubricating value, or else char or vaporize under this higher temperature; and the question of insufficient lubrication and consequent deterioration is a serious one.

While the fluid lubricants have been improved, the pasty lubricants or greases have also received much attention. In general these greases are compounds of a fatty acid with a base. The result of this combination is a soap which may be soluble or insoluble. This soap is dissolved or blended with more or less of lubricating fluid according to the stiffness desired. The primary object of these compounds is to produce a lubricant of greater body than any oil and which can be used in loose or open friction places.

Solid lubricants are often used with these, the greases serving as the vehicle or carrier whereby the dry lubricant is conveyed to the place of service. As a rule, these pasty lubricants are not adapted to cylinder lubrication. The action of the heat and vapor in the cylinder breaks up the compound, leaving a charred or solid residue which is liable to clog the cylinder ports.

Of the solid or ory lubricants, graphite has already been mentioned as the most remarkable. The others worthy of mention are tale, lycopodium powder and mica. A solid or dry lubricant must be fed in the form of a powder. Having no means of feeding such a lubricant conveniently, yet appreciating the value of its low coefficient of friction, many inventors have attempted to use this mineral by mixing it with other substances to form part of the journal bearing itself. Over 125 patents for journal-bearing composition, etc., have been issued with graphite forming the principal anti-friction part of the composition. Lately, many

patents have been issued for various types of lubricators which were designed to feed graphite or some mixture of graphite into the cylinder or on the bearing. Some of these are good, but lubricators are mostly designed to feed only lubricants, and this is the reason why the use of graphite has not been much more extensive, and why it is used sparingly and in cases of trouble.

Many and carefully conducted experiments in the laboratory with Prof. Thurston's testing machine, and experience in shops, have shown that for the highest usefulness the flake must be of a certain size and dressed perfectly pure. Graphite never occurs of the proper size and purity for use. Its natural impurities contain substances fatal to anti-friction purposes. Its proper selection, sizing, and perfecting for lubricating purposes is a matter requiring large skill, much machinery, and great experience. The difference between a perfectly pure graphite and one almost pure, but still totally unfit for lubricating, cannot be detected by either sight or touch.

It is recommended dry, for steam and air cylinders, mixed with grease for heavy bearings, and mixed with oil for light bearings. On being applied to a bearing it readily coats the surface with a shiny, unctuous veneer. These surfaces then slide on each other with very little friction. On being applied to heated bearings the graphite soon fills up any inequalities of the bearing surface due to cutting, abrasion, etc., making them smooth and even, after which the bearing soon cools down. It is equally useful for wood or metal surfaces; in short, in all cases where friction exists. If the bearings are loose enough for the introduction of this thin flake graphite, it will prevent heated bearings, cool those already heated, and reduce friction better than anything else. In all cases where the service required of a lubricant is very severe, graphite will be found specially useful, as in mill steps, gears, heavy bearings, bed plates, etc.

Sometime ago W. F. Downs, of Jersey City, N.J., conceived the idea of extending the use of graphite by making a graphite mixture that would feed through the ordinary type of lubricators. Recent tests of this mixture in some of our most important engine building shops have shown this attempt to be successful. MONOGRAPH ON GRAPHITE

Lubricant	Quantity in Milligramms	Total pressure in pounds on bearing	Temperature at the close	Average coefficient of friction	Time in minutes till the btaring "squealed."	Total number of feet friction surface travelled.
Best sperm oil	335	180	240	. 0555	11	7,198
Graphite mixed with enough water to distribute it over bearings. Graphite mixed with tallow	120 335	180 120	234 340	.0596	30 38	19,635 24,216
* United States Mineral Resources. 1883-84. page 918.	ources, 1883-84	page 918.				

The idea of the suspension of graphite in oil is not at all new, but still seems paradoxical. If it can be accomplished, it certainly solves the question of feeding graphite as a lubricant into many places, where its peculiar properties make it specially valuable. Graphite, by reason of its specific gravity, which is usually given at 2.3, tends to sink rapidly, even more rapidly than other substances of equal specific gravity, on account of its smooth, crystalline surface and low coefficient of friction. Solid particles do not sink as fast in oil as in water, on account of the viscosity of the oil.

If the particles of graphite be coated so as to have viscous faces, and the sizes of the particles be such that the ratio of extent of surface to weight be great, then the combined cling of the coated particles and of the fluid oil offers a force resistant to motion and if this be equal to or greater than the force of gravity the graphite will be distributed or suspended in the oil. This, it is claimed, is done in the manufacture of the compound or mixture of graphite and oil mentioned and the use of graphite particularly as a cylinder lubricant will be largely increased by its success.

In lubricating work the purity and uniformity of the graphite are very essential characteristics. Any impurities present would either cause an oxidation, or hardening or gumming of the oils and greases with which the graphite is mixed, or would be of a gritty character damaging the metal surfaces to be lubricated. The impression prevails that a flake graphite is the only satisfactory form of graphite for lubricating work. This is easily explained and is due to the fact that not all graphites are good lubricants, and that the only natural graphites possessing proper lubricating qualities, and great purity are of the flake variety. Tests have shown, however, that finely powdered graphites, providing they possess the necessary lubricating qualities are equally satisfactory.

In mines, graphite lubricants are used with great advantage. When drills are assembled after cleaning, it is a good plan to apply a graphite lubricant made principally of fine flake graphite, to the moving parts; it imparts a desirable smoothness to the operation of the machine, as well as allowing the quantity of oil used to be considerably reduced.

Dry graphite without any admixture of oil or greasy substances is used for lubrication in air compressors, which contain only dry air. These machines take in the graphite freely if the latter is held on the hand near the suction valves, and several

apparatus have already been constructed, which are attached to the cylinder and allow the piston of the latter during each suction period to draw sufficient powdered graphite to lubricate the cylinder. Experiments have also been made with steam cylinders to lubricate them in the same manner, but special experience in that direction has not been recorded.

The simplest but perhaps not the best way to feed engine cylinders as indicated by the Dixon Crucible Company, is to use it in the regular sight-feed oil lubricators, with tallow cup. It will feed through a very small opening without choking or clogging. A teaspoonful can be put in with the oil twice a day and will need no further attention. The flakes of graphite are carried over one by one to the cylinders and form a coating of perfect lubricating properties.

K. Drechsler in Dresden, Germany, has obtained a patent for a graphite lubricator, which consists of graphite and the yoke and white of an egg; both substances are intimately mixed and then dried and finely ground; the mixture is applied in this condition to the slowly moving parts of machinery.

A graphite lubricant\* for fast revolving shafts consits of equal parts of tallow and graphite.

Another for railroad carriages consists of 36 parts of tallow, 9 parts of pigs grease, nine parts of palm oil and 2 parts of graphite.

### FOUNDRY FACINGS.

The last twenty years have witnessed a great improvement in the foundry practice. From the comparatively light, simple and rough castings of the earlier days, the time has been reached when immense castings are of frequent occurrence, in which the work is often elaborate and the finish is very high; in fact castings either light or heavy, smooth or rough, plain or ornate, are now produced at will, of common, chilled or malleable iron, steel, etc., and all of these different conditions require different services of the facings. Instead of the simple hardwood charcoal dust of earlier times, facings of soapstone, "black lead" or graphite are now used. In all cases where the work is severe, graphite is the constituent, which gives the body "the sleeking and peeling" properties to the facing. The use of graphite for this purpose has increased rapidly with the demand for better and finer foundry work.

<sup>\*</sup> Donath, Der Graphit, page 130.

The main advantages of a facing consisting of a pure graphite are according to the Joseph Dixon Crucible Company, as follows:—

Graphite, or plumbago, as a facing, is put on the surface of a mould for the purpose of preventing adhesion of the metal to the sand of which the mould is composed. A graphite facing saves cleaning of the castings, and gives them a far better appearance. When a properly prepared graphite facing is used, the surfaces are very much easier to work, when the castings are sent to the machine shop. Whenever the molten metal burns into the sand of the mould it causes hard spots on the castings, which quickly dull the edges of cutting tools. The reason why graphite does this is as follows:--Graphite is one of the forms of carbon and is a combustible material, and when the molten metal is poured into the mould, the air in the mould and the air carried in by the stream of molten metal furnish oxygen enough to bring about a certain amount of combustion, forming a film of gas between the metal and the mould. When a drop of water falls upon a hot stove it rolls about; the water itself never comes in contact with the hot surface of the stove, being separated by a film of vapor. It is precisely a similar condition which exists in a mould where a graphite facing is used. The outer portion of the facing begins to burn and a film of gas forms between the facing and the iron. This effectually prevents any adhesion of the metal to the sand, and just as long as this gas film exists, no adhesion can possibly occur. A proper facing will adhere perfectly to the sides of a mould, and will burn and form gas in a slow and regular manner that is—burn just enough to furnish this little film of gas referred to above. We want to form the gas, but to form only the least possible amount of it; and, at the same time, this gas must be formed during the entire time, that the metal is in the fluid condition.

Another point following this is the fact that two bodies cannot occupy the same space at the same time, so that in using a cheap facing, which burns fast and gives out a large amount of gas, this gas is liable to become pocketed inside of the mould, and so prevent the molten iron from filling the mould. This is likely to cause what are called "cold shuts" in castings. The ordinary cheap coal facings are very apt to act in this way. Some facings are not sufficiently adhesive to the sand mould surface. The hot iron, coming in contact with them, immediately dries out the sand, and, if an unsuitable facing has been used, it will run before the metal and leave the mould surface bare.

The conditions which exist in founderies are so various, that it would be quite impossible to make one facing suitable for all purposes.

From the above description it will be noted that a facing possesses two principal qualifications: adhesion and combustibility and as to whether a facing is good or poor, depends entirely on whether these two qualities are properly balanced or not. A facing which runs before the metal is no better than one which adheres, but burns up before the metal chills.

#### GRAPHITE PAINTS.

A very important use for graphite is found in its application as a protective coating, especially for iron structures. Thousands of important steel structures all over the world are protected from corrosion with a good graphite paint, and durability records in different climates, under severe conditions of service, prove it to be the best and most economical protective paint in use. A graphite paint, in which pure ingredients are used, covers perfectly more surface to the gallon and protects from rust longer than lead, metallic, asphaltum or composition paints.

The main ingredients entering into the composition of a good graphite paint are: A hard flake graphite, pure silica, and pure double-fire boiled linseed oil.

The flake graphite retains its laminated crystalline structure to the finest degree of pulverization, thereby better protecting the binding material from destruction than the soft, cheap amorphous forms of graphite.

A proportion of pure silica is added as a filler; the silica graphite pigment remains unaffected by heat, acids, gases or chemicals.

The linseed oil gives a tough, leather-like, elastic coating, unequalled for its wearing qualities. This oil and silica graphite pigment produces a preservative coating, which cannot be otherwise than durable and economical.

Many years ago the use of graphite for paints was advocated, and experiments showed the value of the suggestion. Unfortunately, the success of these experiments has since caused a flooding of the market with a great number of so-called graphite paints, which really contain no true graphite.

Many so-called "carbonaceous schists" are used in the manufacture of paints; they consist of silicious materials mixed

with various amounts of carbon, which gives to the whole a black, graphite-like appearance. These rocks are crushed to a fine powder, which is then mixed with oil. Specious arguments are at the same time advanced with the object of convincing the public that it is a mistake to use pure graphite in paints.

It must be admitted that a mixture of pure graphite and oil is a slow dryer, and does not produce a coating with sufficient rapidity to satisfy the average consumer, who is inclined to prefer a rapidly drying paint, but with poor covering qualities, to a slow drying article; the latter will, however, give the maximum and most permanent protection from corrosion. It must be evident to everyone that the objection above referred to can easily be overcome by the addition of suitable driers to the paint, and that one of the first requisites of a good paint is the purity of its constituents.

The drier can be selected to meet the requirements of each particular case, and the degree of drying can be regulated at will. Many graphites or so-called graphite pigments are either byproducts or low-grade ores, in both cases too impure for other purposes. They vary considerably in quality, and much care must be exercised in the selection of a good graphite for paints.

In special cases, it is desirable to use pure graphite for protective purposes, as in the case of the shields for the cables of the new East River Bridge in New York. The protective material used in this case is a mixture of graphite and oil. The protection of the cables of a great bridge like this from corrosion is a matter of the greatest importance, so none but the purest materials can be used.

Good silica graphite paint weighs 9.75 lbs. per gallon, and has a covering power of 500 to 600 sq. ft. per gall. for an even coat of metal surfaces and about 350 sq. ft. per gall. on wood. The smoothness of the pigment saves materially in cost of labor and brushes in applying. The cost of the paint, ready mixed for use, is about 120 doll. per barrel of 50 gall. At this rate the cost per sq. ft. is about 0.48 cents for metal and 0.52 cents for wood surfaces. Its practical use in all climates has shown it to be unequalled as a protector of wooden or metal surfaces. It will protect steel viaducts and bridges from corrosion for five to fifteen years, iron and tin roofs for ten years, and steel smoke stacks from one to five years. It resists for years, the rust forming elements, rain, snow and heat of the sun. It is the best protective coating for surfaces subject to sulphur fumes.

#### VARIOUS USES OF GRAPHITE.

Graphite finds an important application on account of its great electrical conductivity in electrotyping. A fine film of graphite over objects made of gypsum, imparts to the latter a great conductivity without causing any deformity in the appearance of the article produced. For this purpose very pure graphite of a fine scaly quality must be used.

As a polishing material, graphite has been known for a great many years. In the manufacture of gunpowder the little pellets are generally polished with graphite, while Fadejeff\* recommends it also for the packing of gun powder to render its combustion more difficult. For this purpose the bottom of the barrel to be used for packing is covered with a layer of two inches of a finely powdered mixture of graphite and charcoal, while the gunpowder is also mixed with the same and placed in the barrel and covered again with a two inch layer of the material. It is claimed for this mode of packing that the contents of the barrel may be ignited and will quickly burn away without explosion. Before using, the pellets of the gunpowder are separated from the graphite and charcoal by sifting.

For the manufacture of stove polish, graphite is extensively used, and, according to statistical data at the disposal of the writer, fully 15% of the total production of graphite is used for this purpose. There are two kinds of stove polish in the market: one is in a dry state and represents as a rule the refined (washed) graphite in lumps sold by the pound to the consumer as in Europe. and the other consists of a thick paste, in the main parts consisting of benzin, some oil or soap and impalpable powdered graphite.

That graphite can also be used as a preventive of boiler scale is perhaps not generally known.†

A very small quantity of graphite added to water used for boilers will prevent scaling, and if the boiler already has scale it will not only prevent further deposition, but the minute particles of graphite will penetrate the old scale and soften it, so that it will drop to the bottom and can be taken out inexpensively. This assertion is based upon facts. Having used water taken from the mine, which was only slightly discolored by graphite, the softening of the scale was accidentally discovered.

<sup>\*</sup> Eng. & Mining Journal, 1903, page 677. † Donath Ibid, page 131.

The water apparently contained nothing else which could have produced the same result. The water was used probably two months before the discovery was made.

Graphite is used in a stove or boiler cement consisting of 40 parts of powdered graphite, 40 parts of barytes, 20 parts of ground fire brick, 1.5 parts of lime, and 24-26 parts of varnish. This composition, which hardens, sometimes makes a quick repair for broken stoves, range linings or leaking boilers.

The Morgan Crucible Co., Ltd., and C. W. Speers, London,\* England, patent 9875, May 10th, 1905, describes a process wherein plumbago, crystalline or flaked, such as that from Ceylon, is ground to pass through a 100-mesh sieve and is compressed in moulds, under pressure of not less than 20 tons per square inch, with suitable direction of the plane of pressure to the purpose for which the blocks are intended.

For commutator brushes, for instance, the lines of stratification should run in the direction of flow of the current, while for wearing surfaces the planes of stratification should be at right angles to the wearing surface.

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<sup>\*</sup> Eng. & Mineral Journal 1905, II 1017.

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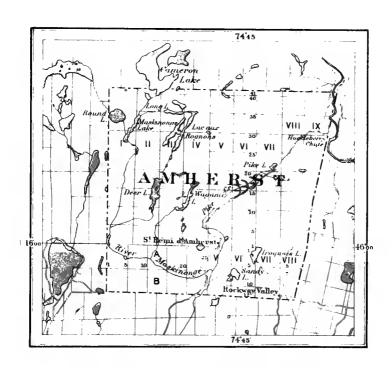
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1907



### GRAPHITE OCCURRENCES

IN AMHERST TP., QUEBEC

Scale: 35000 or 3.95 miles to one Inch

1 2 3 4 5 6 7 8 9 10

Miles

James White, F.R. G.S., Geographer Dept. of Interior

Accompanying Monograph by Fritz Cirkel, M.E. Eugene Haanel, Ph. D., Director of Mines

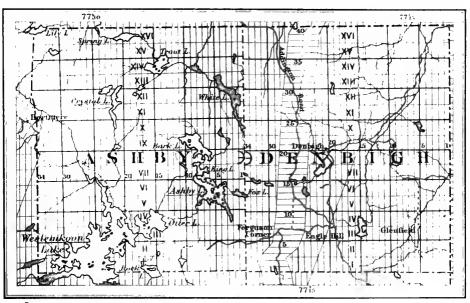
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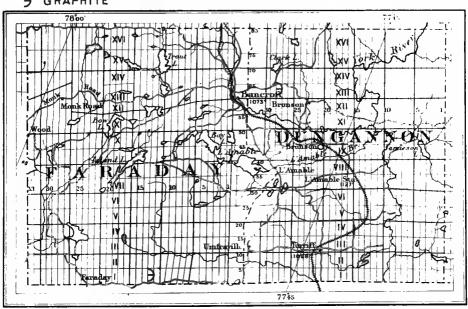


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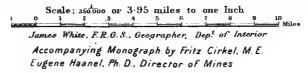


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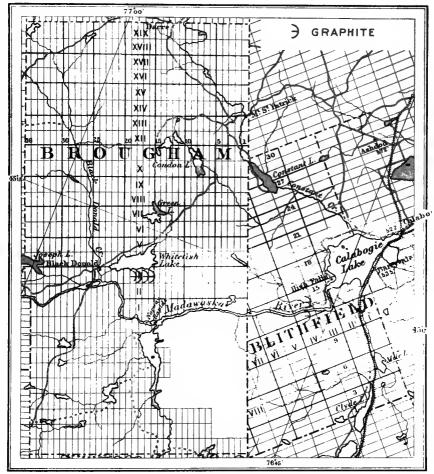
#### GRAPHITE OCCURRENCES

IN ASHBY, DENBIGH, FARADAY AND DUNGANNON TPS., ONT.



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# GRAPHITE OCCURRENCES IN BROUGHAM AND BLITHFIELD TPS.. ONTARIO

Scalc; 350,000 or 3.95 miles to one Inch

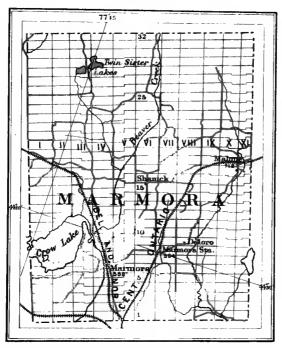
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) GRAPHITE

### GRAPHITE OCCURRENCES

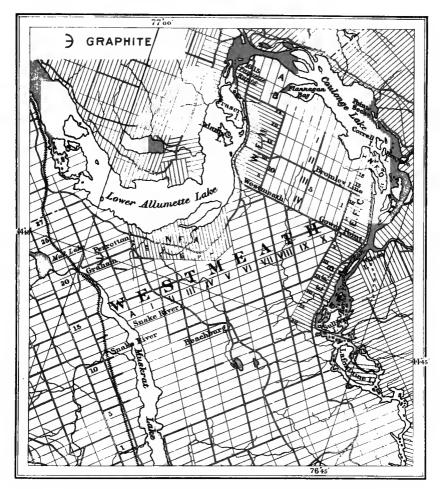
IN MARMORA TP., ONTARIO

Scale; 350,000 or 3.95 miles to one Inch

1 2 3 4 5 6 7 8 9 1

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### GRAPHITE OCCURRENCES

IN WESTMEATH TP., ONTARIO

Scale; 35,000 or 3.95 miles to one Inch

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